PETROLEUM PROCESSING

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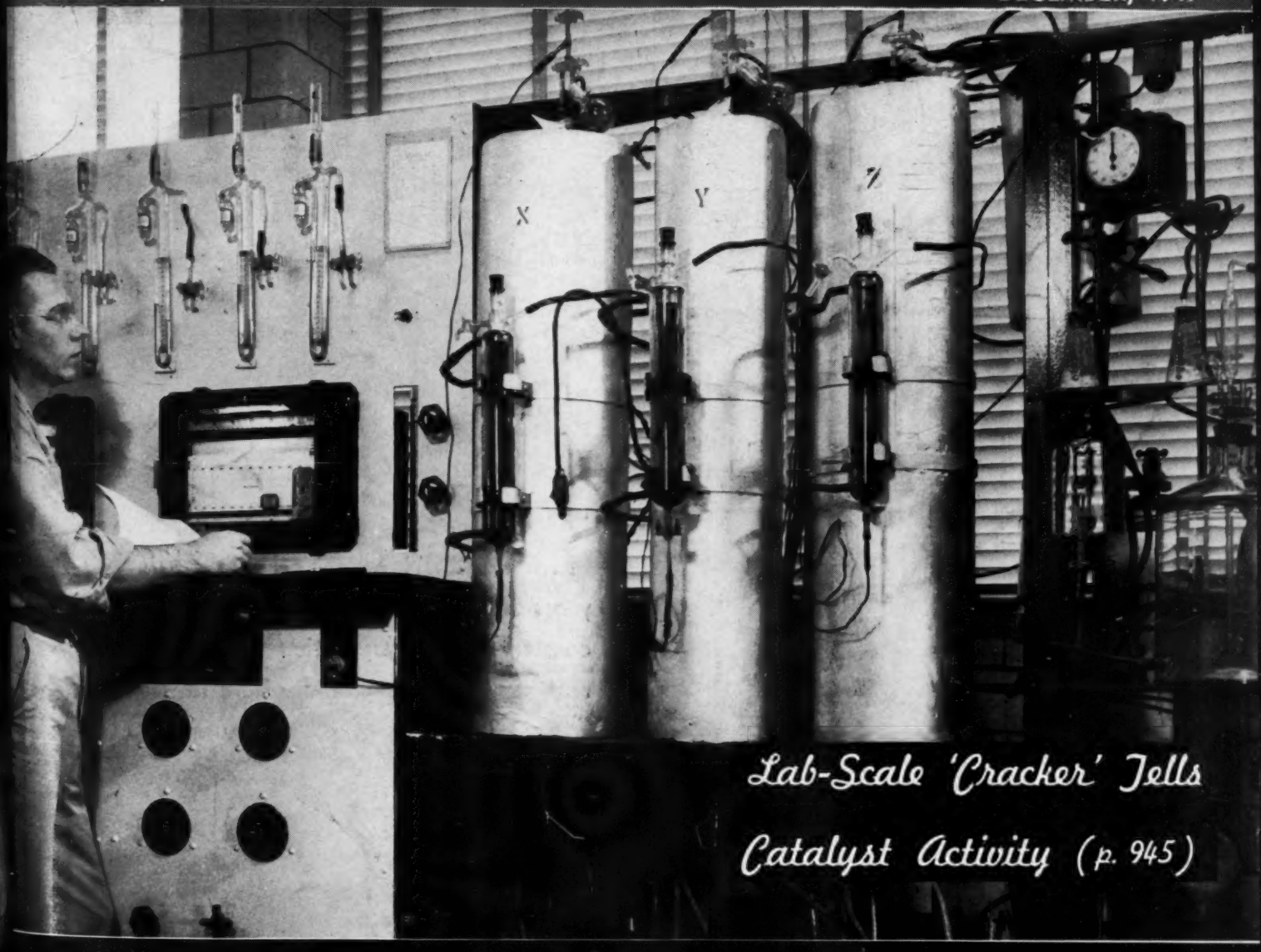

Lab-Scale 'Cracker' Tells Catalyst Activity (p. 945)

FORMERLY MONTHLY TECHNICAL SECTION OF NATIONAL PETROLEUM NEWS

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COVER STORY

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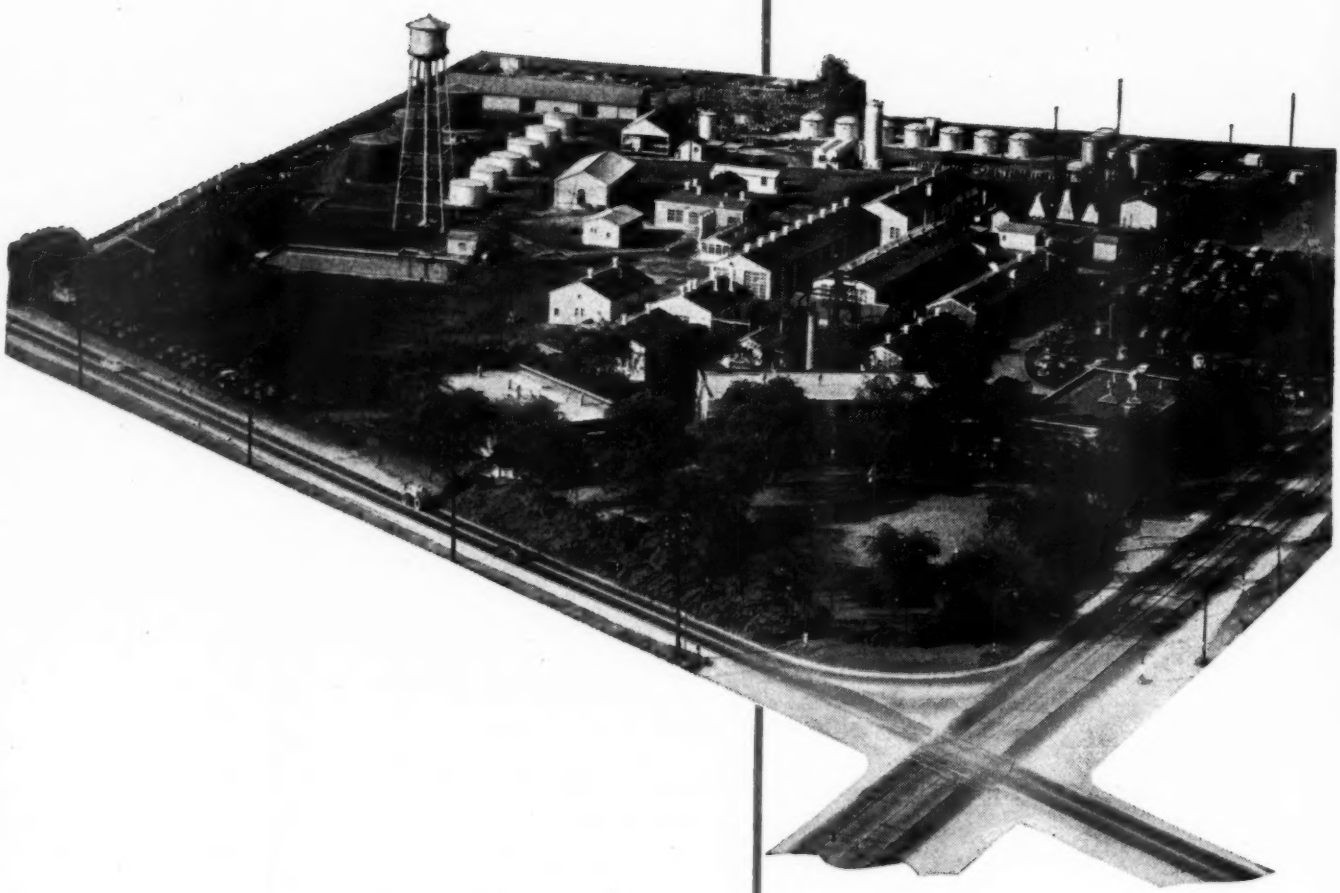

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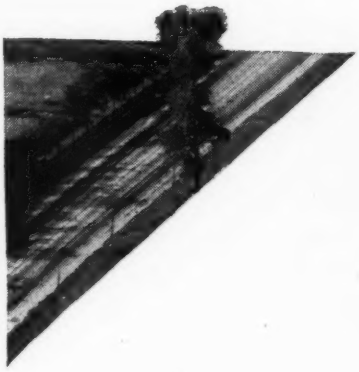

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What's Happening!

DECEMBER 1
1947

HIGHLIGHTS *Gulf Oil Corp. announces $20,000,000 refinery improvement program. . . . Sun Oil Co. to consolidate its research facilities in new Science Center. . . . Latest estimate of 1948 total petroleum demand in U. S. is 6,104,000 b/d, about 4.5% over 1947 demand. . . . API urges Congress to legislate defining authority of Federal Power Commission as not including right to regulate production or gathering of natural gas. . . . Harriman Report sees "world-wide" oil shortage as principal limiting factor in supplying requirements of European nations under Marshall Plan.*

Plants Building New facilities under construction at the Robinson, Ill., refinery of Ohio Oil Co. include a Fluid cracker, two-stage distillation unit, gas recovery unit, UOP catalytic polymerization unit and Girbotol gasoline treating plant. Crude charging capacity is to be doubled to 30,000 b/d. Universal Oil Products Co. is designing and engineering the new units. Arthur G. McKee & Co. is general contractor. J. F. Pritchard & Co. is building some of the utilities.

Sun Oil Co. has started construction at Newtown Square, about 18 miles west of Philadelphia, of a new physical laboratory as first step in creating a science center in which will be consolidated research and development work now carried on at the Marcus Hook refinery and the Norwood, Pa., laboratories.

Gulf Oil Corp. announces a $20,000,000 refinery improvement program, major construction work to start next spring. New catalytic cracking units will be installed at Toledo and Cincinnati and output of the latter refinery will be increased 25%. A new "poly" unit will be built at Cincinnati. A new laboratory will be built at Toledo and the Cincinnati laboratory enlarged. Major construction work will be by E. B. Badger & Sons Co. and M. W. Kellogg Co. Chicago Bridge & Iron Co. has the contract for relocating and rebuilding the tankage.

Catalytic Construction Co. has started work of inspecting, repairing and testing the Houdry unit of Petrol Terminals Corp., recently purchased from WAA. Repairs and catalyst change to the Houdry unit at Socony-Vacuum Oil Co.'s refinery at Buffalo are being made by Catalytic Construction Co.

Plans are being made for modernizing and enlarging the 3000 b/d topping plant of Comet Refinery Co. at New Brighton, Minn., recently acquired by Northwestern Refining Co.

Sinclair Refining Co. has awarded the contract for a 270-mile products line from Houston to Arlington, near Fort Worth. A terminal will be built at Bryan, where the new line will connect with the system Sinclair has been building this year from Corpus Christi. The line to Fort Worth is to be completed early in 1948.

Foreign Petrocarbon, Ltd., has announced plans for new 20,000 b/d refinery near Manchester, England. Plant will be built adjacent to facilities now building by Petrochemicals Ltd. and its operations will be tied in with those of Petrochemicals and also of Manchester Oil Refinery, manufacturer of white oils. Petrocarbon facilities will include 5000 b/d catalytic cracker, reforming unit and lube manufacturing facilities.

British Petroleum Chemicals, Ltd., owned jointly by Anglo-Iranian Oil Co., and Distillers Co., Ltd., is planning to build petroleum chemicals plant adjacent to Anglo-Iranian refinery at Grangemouth, Scotland, at a cost of $22,000,000. Anglo-Iranian is planning $30,000,000 expansion of the Grangemouth refinery.

Two West Coast oil companies, Honolulu Oil Corp. and Los Nietos Co., have declined to participate in the American Independent Oil Co., organized by a group of independent concerns to carry on operations in foreign oil fields.

Explain Product Allocations Socony - Vacuum Oil Co. publicly announced it would allocate motor and aviation gasoline, naphthas and Diesel fuel supplies to dealers and direct customers in its eastern marketing territory during November and December, on a basis 4% less than quantities delivered in same months of 1946. Company also took prominent newspaper advertising space to tell its allocation policy and advise public what they could do to relieve the shortage.

'48 Demand and Supply Revised estimate of Bureau of Mines for total U. S. demand for petroleum in 1948 is 6,104,000 b/d average, including 407,000 b/d for export. This is about 4.5% above total 1947 demand as estimated. Crude runs to refineries are estimated at 5,265,000 b/d average, little over 4% greater than for current year.

Domestic demand for motor fuel for '48 is now estimated at 840,000,000 bbls., over 6% higher than in '47; for distillate fuel and kerosine 438,000,000 bbls., 12% higher; for residual fuels 517,000,000 bbls., less than 1% higher than '47 rate of consumption.

Total production of crude and other oils in U. S. is estimated for '48 at 2,075,300,000 bbls., nearly 5% larger than for current year. Total imports, crude and refined, are estimated at 166,800,000 bbls., nearly 6% above current year.

Synthetic Rubber Synthetic rubber is now being produced with 35% more wearing quality than natural rubber and further improvements may be expected through present research programs, George P. Bunn, vice president Phillips Petroleum Co. and manager of its butadiene plant at Borger, Texas, told the synthetic rubber sub-committee of the House armed services committee. Bunn and officials of Humble Oil & Refining urged government legislation to continue requiring use of synthetic rubber in tires and that the synthetic rubber plants

be made available to private operators through sale or lease.

European Aid Report Studied National Petroleum Council's committee on the Marshall Plan has created a subcommittee headed by Howard Page, economist, New Jersey Standard, to analyze the Harriman Committee report on the European recovery aid program insofar as it relates to petroleum requirements.

The Harriman report states it is unlikely the presently estimated petroleum requirements of the 16 participating European nations for 1948-51 can be met in full because of a "world wide" shortage of oil. The shortage may range from 5-10% below requirements in 1948 to 10-15% in 1951 and concludes that the European nations must therefore readjust downward their planned levels of imports and consumption.

The Harriman report also regards it unlikely the U. S. can supply the 16 countries with the $588,000,000 in petroleum equipment they say they need, but urges that maximum aid be extended in this regard.

POL Reserve API Defense Services Committee is naming committees for each of the 6 Army areas in continental U. S. to promote and aid individual oil companies in organizing petroleum reserve units as sought by armed services. Committee will also ask all companies to report on their personnel who have reserve status.

General Resolution urging prompt passage by Congress of law defining authority of Federal Power Commission as not including right to regulate production or gathering of natural gas adopted by API directors at Chicago annual meeting.

Ashland Oil & Refining Co. has purchased from WAA the government-owned "avgas" plant near Catlettsburg, Ky., which it operated during the war. Purchase price was $2,150,000. Facilities include TCC cracker, feed preparation unit, alkylation and isomerization units.

This country is cutting by one-half import duties on gasoline, naphtha, paraffin and paraffin wax and unfinished petroleum oils, in return for certain tariff concessions by other countries which signed 23-nation trade agreement in Geneva Oct. 30. Provisional application in U. S. is by presidential proclamation. Crude oil import duty is not affected.

Technical The need for an SAE 5 or 5-W motor oil to meet service conditions of atmospheric temperatures of —10°F. and below was advanced by automotive technologists at the SAE Fuels and Lubricants meeting in Tulsa. The oil should have the same viscosity at —20°F. that the 10-W oil has at zero. Use of a 10-W oil diluted with kerosine is not adequate to meet driving conditions at extreme low temperatures, it was said.

At the same meeting another speaker suggested the use of suitable additives of the oil-soluble polymethacrylate type with appropriate stocks, to secure enough Viscosity Index improvement to permit double branding in commonly used SAE grades, thus simplifying storage and handling problems.

Standard set of designations for automotive gear lubricants adopted by API Lubrication Committee including: regular type gear lubricant, worm type gear lubricant, mild type extreme pressure gear lubricant, multi-purpose type gear lubricant. Proposal for lubricant recommendations for automotive gears was tabled because of divided opinion of committee membership.

Promoting Oil Research Shell Oil Co., Inc., Shell Development Co., Shell Chemical Corp., Shell Union Oil Corp. and Shell Pipe Line Corp. have jointly established 44 fellowships at 36 U. S. colleges, the total grants amounting to about $76,000 a year. The fellowships will be awarded by the schools, their purpose to be to promote graduate study in branches of science which affect the petroleum industry.

Product Supply, Requirements Supplies of gasoline, kerosine and distillates east of the Rockies will fail to meet estimates of demand by about 5% during the next 5 months, if the deficiency is spread uniformly among products, John W. Boatwright, manager of sales research for Standard Oil Co. (Indiana) told the API meeting. If refiners recognize the composite yield of the three products as 65.2% of crude runs, which was achieved last winter, and adopt yields or 39.8% on gasoline, 7.4% on kerosine and 18.0% on distillates, the deficiency forecast for a normal winter would be spread uniformly among the three products. This is desirable to show public the industry is being fair to all classes of consumers.

Looking farther ahead, Fred Van Covern, API statistical director, said he doesn't look for any national shortage of gasoline next summer but that in certain areas, particularly District 2, the companies will have to continue to allocate supplies as at present.

Top Names In the News Dr. William M. Burton, retired former president of Standard Oil Co. (Indiana), received the API Gold Medal for Distinguished Achievement in recognition of developing the first commercially successful cracking process.

Dr. John R. Bates, formerly assistant to Sun Oil Co.'s vice president in charge of manufacturing, is director of its new independent research department.... James Tanham, vice president The Texas Co., is attending the United Nations conference on trade and employment in Havana as a representative of the International Organization of Industrial Employers.

J. R. Corbett, Cato Oil & Grease Co., Oklahoma City, is the new president of the National Lubricating Grease Institute; B. G. Symon, Shell Oil Co., New York, is the new vice president.

Carl E. Cummings, superintendent of The Texas Co.'s research and development laboratories at Beacon, N. Y., and Charles G. McIlwraith, physicist in the Beacon laboratories, have received the Order of the British Empire, Degree of Honorary Officer, for their wartime services.

T. M. Martin, executive vice president of Lion Oil Co., succeeds Col. T. H. Barton as president. Col. Barton was elected chairman of the board.

Leonard F. McCollum, formerly co-ordinator of producing activities for Standard Oil Co. (N. J.), has been elected president of Continental Oil Co., succeeding Dan Moran, who resigned because of ill health but will continue in consulting capacity with the company. . . . . **John W. Brice** is co-ordinator of producing activities for New Jersey Standard, succeeding L. F. McCollum.

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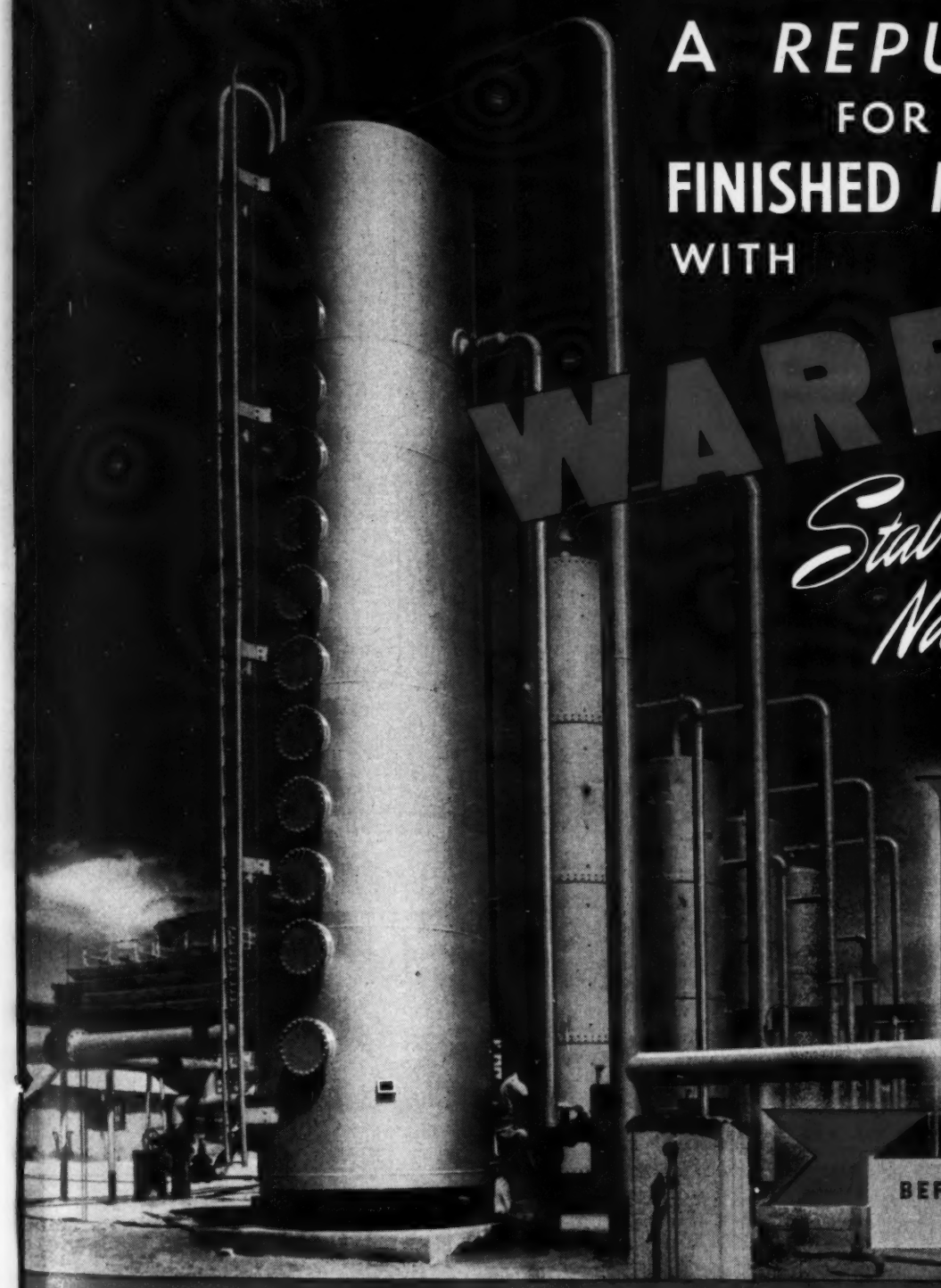

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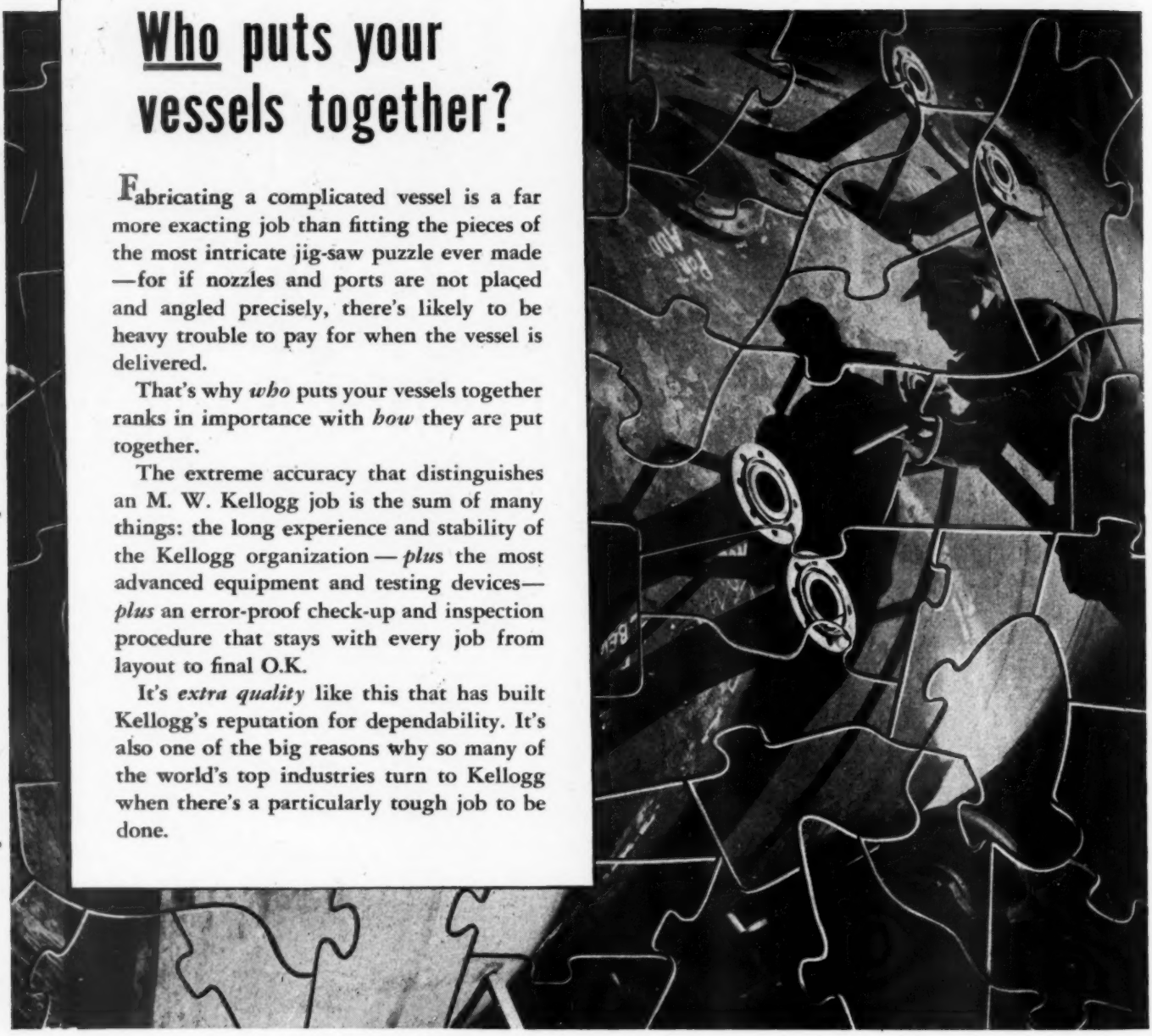

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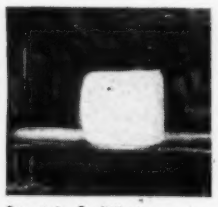

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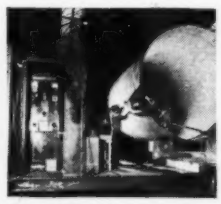

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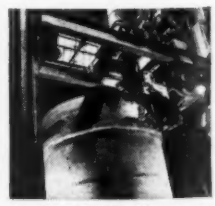

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TOMORROW

... IN PETROLEUM TECHNOLOGY

Current technological and economic developments discussed here in the light of their future bearing on petroleum refining operations include:

- **More economic data on synthetic fuels available.**
- **Atomic power plants still a long way off.**
- **Public Library Establishes Research Group**
- **Detergent promoter developed for fabric washing.**

More Economic Data on Synthetic Fuels Available

AS MORE and more organizations concentrate attention on the synthesis of petroleum substitutes, an increasing amount of economic information is becoming available for purposes of comparison and evaluation.

For example, Dr. W. C. Schroeder, chief of the Bureau of Mines' Office of Synthetic Liquid Fuels, recently told the oil subcommittee of the Senate's special small-business committee that while gasoline can be produced from natural gas, via the Synthine process, at a cost competitive with present-day petroleum, oil from shale would cost $2-$2.50 per barrel for a product comparable only with $1-$1.40 petroleum.

Schroeder stated, as is common knowledge, that petroleum products of any type or grade can be made from coal and that the cost of gasoline so produced would be 3-5 cents per gallon greater than that made from petroleum. A coal hydrogenation industry capable of producing 1,000,000 barrels of gasoline per day would require 225,000,000 tons of coal per year. About 150,000 men would be needed in mines and plants, and construction of such facilities would require 10,000,000 to 11,000,000 tons of steel.

According to a recent item(1) the Standard Oil Development Co., following wartime research for the Australian Government on the production "of marine fuel from Australian oil shale by fluidized solids techniques," concluded "that the method was novel and sound, and that costs would be almost totally governed by the price of raw shale as delivered to the retorting plant. It was also their opinion that oil shale processing in the United States might to some extent become competitive with other methods of crude oil production in the not-too-distant future —that is, if the cost of the mined product can be cut to around 50 cents per ton."

In commenting on the use of fluidized solids technique in the production of oil by the Synthine process, Dr. H. H. Storch of the Bureau of Mines recently told the Philadelphia Section of the American Chemical Society that pilot-plant tests have demonstrated that a Synthine plant using this process may operate continuously for as long as 1,600 hours. Gasoline, easily refined to 75 octane, would be the chief product, along with minor quantities of Diesel oil and chemicals.

In discussing the Bureau of Mines' Synthine process in which oil, introduced along with the gas charge, evaporates and thus provides temperature control through heat absorption, Storch stated that at present this fixed-bed process is only two-thirds as fast as the fluidized catalyst process and requires one-third more steel. Further research is expected to improve its efficiency, however.

James A. Lee, managing editor of *Chemical Engineering,* reports that, for Jersey Standard's fluid catalyst variant of the Synthine process, a plant "large enough to produce 8,300 b/d. of gasoline giving an ASTM unleaded number of about 80 after mild treatment and 1,050 b/d. of gas oil . . . would cost about $35,000,000, exclusive of the investment for recovery of oxygenated compounds."(2)

Incidentally, "attracting attention are reports that Carbide and Carbon Chemicals Corp. is pursuing experiments in the underground gasification of coal at Charleston, W. Va., and that these have progressed beyond the stage reached by the much-publicized Alabama Power Co. and the Bureau of Mines project at Gorgas, Ala. Recently, Carbide purchased 30,000 acres of coal land in the Charleston area."(3)

All this activity in research on synthetic liquid fuels bodes well for the ultimate future. However, the petroleum industry must remember to point out again and again, in its public releases, that petroleum substitutes cannot be produced in quantity at a moment's notice and that a large-scale synthetic fuels industry, were it to be started now, would be uneconomic even before completed, both because of technical obsolescence and present insufficient process development. Continued research is therefore more than justified, especially since present technical knowledge, at least as regards the Synthine process, gives every indication that future costs may be lower and product qualities higher. Such, alas, is rarely the case for petroleum itself, these days.

Atomic Power Plants Are Still A Long Way Off

THOSE science writers who in late 1945 predicted the near-future end of petroleum usage for fuels, because of the advent of atomic energy, are now more than quiet on this subject. First of all, they ignored the fact that most petroleum fuels are "burned" because their liquid state renders them particularly portable and useful, while atomic energy "power pills" would require carrying tons of shielding materials even if their technical application to transport vehicles were perfected. Secondly, they assumed that technical developments in the field of applied atomic (nuclear) science would progress at an accelerated pace, which has hardly been the case; research of any type costs money, and "the war is over"—but the next one may come soon.

As *Business Week* recently put it, "new (atomic power timetable) estimates reflect a revised allocation of manpower by Atomic Energy Commission. Military work gets top priority."(4) Admiral William S. Parsons, director of the Navy's atomic defense group, recently told the Socity of 1812 that: "(1) it will take 10 years to develop high temperature nuclear reactors for special purposes; (2) large-scale output of industrial atomic power is a generation away."

Only as recently as last March, however, "Parsons predicted: (1) experimental power pile operation in 1948; (2) solution of engineering problems in five years; (3) development of atomic power to a point of practical use in the early 1950's."

The new motif—delay—was also sounded in a recent speech made by David Lilienthal, chairman of the Atomic Energy Commission. Accord-

(1) Anon., *Industrial and Engineering Chemistry* 39, No. 10, Suppl. p. 10A (1947), "I & E C Reports—Oil Shale."

(2) Lee, James A., *Chemical Engineering* 54, No. 10, 105 (1947), "Esso's Fluid Catalyst Modifies Fischer-Tropsch."

(3) Anon., *Chemical Industries* 61, No. 3, 371 (1947), "For Your Information."

(4) Anon., *Business Week*, No. 942, 6 (1947), "Washington Outlook."

ing to Lilienthal, "Our judgment is that no one should delay sound and economical additions to power supply, whether by fuel-generated electricity or water power, because somewhere in the future atomic energy will come on the scene as an additional source of supply. There will certainly be ample time to make whatever adjustments may be needed."

Lilienthal's atomic-power timetable is much like Parsons' (for obvious reasons): (1) "production of electricity on a pilot-plant scale of 1,000 kw. or so—18 to 20 months from now;" (2) "large-scale production of electricity in a 'useful practical demonstration plant'—eight to 10 years from now;" and (3) "atomic supply of 10%-20% of the country's power needs—'around two corners.'"(5)

According to this report(5) of Lilienthal's speech, "the major problems in power generation are still at the level of university research rather than engineering investigation." By some, however, although denied by Lilienthal, "partial relegation of power studies to the academic groups is interpreted as a move to free nuclear and chemical engineering talent for the big job of rebuilding the production plants (at Hanford and elsewhere) for maximum output of explosive material for bombs." Moreover, "Lilienthal says he is now stressing that the time scale is long so as to answer European claims that the U. S. is selfishly sitting on a technology which could solve the world's fuel shortages."

Interesting technical and economic data on atomic power have been summarized in a recent article(6) by James H. Lum, executive director of Monsanto's Clinton Laboratories at Oak Ridge. In essence, this article confirms the 1946 economic data of the Baruch report: "(1) Atomic plant—total investment, $25,000,000; investment per kw., $333; operating cost per kwh., 0.80 cent; (2) Coal plant—total investment, $10,000,000; investment per kw., $133; operating cost per kwh. with coal at $7 per ton, 0.65 cent; operating cost with coal at $10 per ton, 0.80 cent."

It is significant, from a petroleum industry viewpoint, to note that three members of a seven-man industry committee appointed by Lilienthal "to speed private enterprise's part in atomic energy development"(5) are prominent petroleum research scientists and executives—Dr. Gustav Egloff, director of research of the Universal Oil Products Co.; Dr. Paul D. Foote, executive vice-president of the Gulf Research & Development Co.; and Dr. Robert E. Wilson, chairman of the Standard Oil Co. (Indiana). It may be expected, then, that the petroleum industry will be readily able to evaluate future developments in atomic power as they occur, although apparently there is no reason to fear that atomic power will relegate petroleum completely to the status of a chemical raw material in the predictable future.

(5) Anon., *Business Week*, No. 946, 24 (1947), "Atomic Timetable."

(6) Lum, James H., *Chemical Engineering* 54, No. 10, 122 (1946), "Engineering and Economics of Atomic Power."

Public Library Establishes Research Group

IN these days of increased attention to the teachings of the technical literature, it is highly pertinent to note the organization of the Industrial Research Service foundation, associated with and organized through the efforts of the Detroit Public Library.

As stated by Charles M. Mohrhardt, associate librarian of the Detroit library,(7) "literature research in the business and industrial fields is carried on, not to increase man's mastery of his environment, but because it pays in dollars and cents to the company or individual who is engaged in such research.

"Most large corporations maintain special libraries, not as frills but as important service units. Even the most extensive of these special company libraries must depend upon the greater resources of a large public library to obtain information required by their corporations," however, and it was here that the Detroit Public Library, noting increased demands on it for business and industrial reports, literature searches, and speedy information service, decided to expand its information research services and to offer them on a subscription basis.

In brief, the plan calls for the creation of the Industrial Research Service, "which will be designed to supply information, bibliographical and documentary service to those firms which are willing to support the maintenance of this service. . . . We recommend that the large corporations subscribe to this service at an annual rate commensurate with the services received. For the smaller firms and those who do not wish to subscribe to this comprehensive research service," the usual free, less extensive library service is still available, or they can "make use of the Industrial Reference Service and be billed for the work performed, on an hourly basis."

"Confidential reports would be restricted to the subscriber who had requested them"; others could be published for general use. Among the services to be offered are: (1) a general reference or fact-finding service, (2) bibliographical service, (3) translation service, (4) literature studies and documentation service, (5) abstracting service (on new publications), etc. Specialists in the fields of service are to be employed full-time or as part-time consultants; these will work with library specialists.

The annual service fee will "be based on the annual expenditures of the subscribing company or, more specifically, upon the amount which is allocated by the company to research, personnel management, marketing, and other comparable programs for which they require library assistance. . . . It is proposed that business and industry underwrite the costs of this program for a three-year period" to insure continuity of staff, etc. For the first year, the Detroit Public Library plans to limit this service to the automotive industry.

The petroleum industry subscribes to a vast amount of research, much of it properly based on information work, and "it is perhaps unfortunate that there is at present so little cooperation between petroleum companies on informational matters."(8) An ideal arrangement might call for the collection of existing information by a central group, the classification of this material under a generally acceptable system, and "the central provision of future information to all who are contributing members."(8)

In any case, the Detroit Public Library's service to the automotive industry can be viewed with careful attention by the petroleum industry and analyzed for adaptation to its own operations.

(7) Mohrhardt, C. M., *Library Journal* 72, 1155-59, 1166 (1947), "Detroit Projects New Research Plan."

Detergent Promoter Developed For Improved Fabric Washing

AS mentioned last month, most synthetic detergents "are not completely satisfactory for washing heavily-soiled cottons" and "do not in general hold an edge for use in soft waters." It is therefore important to note a brief news item(9) which states that the "Wyandotte Chemicals Corp., Wyandotte, Mich., has developed a 'detergent promoter' it believes will cover that gap (the fabric washing market). The firm says it is actually superior to 'fatty acid soaps in detergency on all fabrics, including cottons.'"

Wyandotte, last year, began construction of a plant for the production of petroleum-derived alkyl aryl sulfonate detergents. More details on the above-mentioned "detergent promoter" will be awaited with interest, since agents of this type might very well give added impetus to the already-fast-moving synthetic detergents field.

(8) Weil, B. H. Information Service and the Petroleum Industry, Georgia School of Technology, State Engineering Experiment Station Circular No. 10, 1947. Five pages.

(9) Anon., *Business Week*, No. 50, (1947), "Detergent for Fabrics".

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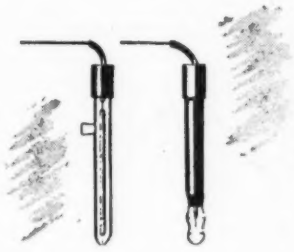

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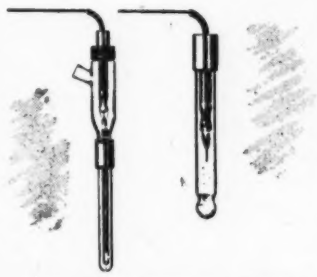
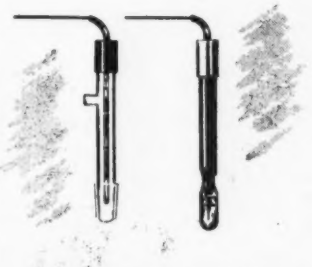
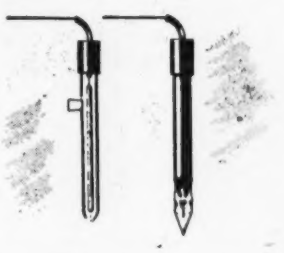


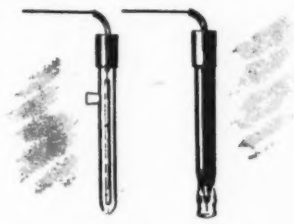
HIGH TEMPERATURE GLASS ELECTRODES. Another development pioneered by Beckman, these electrodes can be used directly in high temperature process solutions to 100° C. (212° F.) without damage. Eliminates special sampling devices and greatly simplifies control of hot processing solutions.



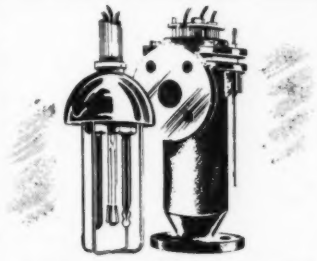
RUGGED DUTY GLASS ELECTRODES. Still another Beckman development, these electrodes are specially designed for direct immersion in tanks and flow lines where abrasion is severe (paper pulps, ore slurries, etc.). Not only abrasion resistant, but also withstand more than 100 lbs. pressure on immersion end.



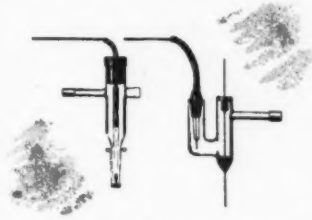
PENETRATION TYPE GLASS ELECTRODES. The pointed application end greatly simplifies direct pH measurements of cheeses, fish, fruits and similar semi-solids where penetration of outer surface is desired for internal pH measurements.



SURFACE TYPE GLASS ELECTRODES. On these Beckman electrodes the application end is nearly flat in order to simplify pH measurements on skin, hides and moist surfaces of materials where pH measurements of outer surfaces are desired.



IMMERSION AND FLOW TYPE ASSEMBLIES. Can be placed directly in flow lines, vats, tanks, etc., to permit direct and continuous pH measurements on process solutions. Far simpler, quicker and more accurate than sampling devices. Can be used with various types of Beckman electrodes.



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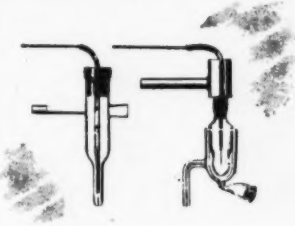

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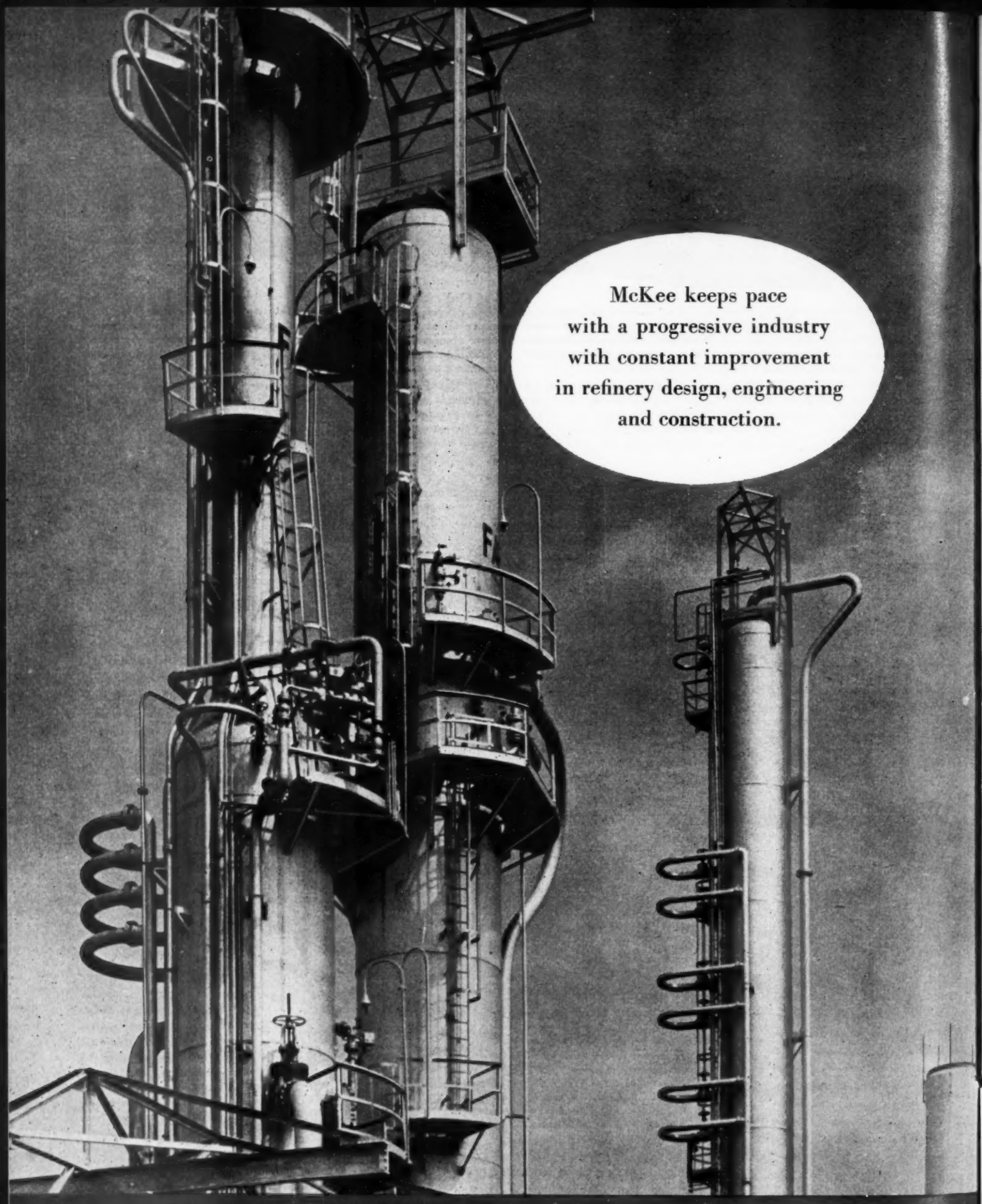

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PETROLEUM PROCESSING
DECEMBER, 1947

Sour Crude Ratio Not Likely to Increase Refiners Are Told at API Meeting

By WILLIAM F. BLAND
Engineering Editor

CHICAGO — The best refining brains from all over the country converged on the Stevens Hotel here Nov. 10-14, to get the lowdown on one of their most pressing operating problems—handling sour crudes.

They learned what the outlook on sour crude supply is, what quality of products are produced from sour crude, and how one company combats the difficult corrosion problems of sour crude processing.

The occasion was the 27th annual meeting of the American Petroleum Institute. One of the three group sessions of the API Refining Division held as part of the annual meeting was devoted to "Sour Crude Processing" and included three papers on the subject.

Supply Situation Not Rosy

On the sour crude supply situation, as presented in a paper by H. M. Smith and O. C. Blade, of the U. S. Bureau of Mines Petroleum Experiment Station, Bartlesville, refiners learned that:

1—In 1946 the total production of high-sulfur crude oils reached a new high, and the ratio of high-sulful crude oils produced to low-sulfur crude oils was also an all-time peak value.

2—The relative sulfur content of estimated reserves of the country, however, show approximately the same pattern in distribution as do the sulfur contents of crude oils currently produced, which would indicate that, until new fields of considerable magnitude are discovered, the ratio of high-sulfur to low-sulfur oils should not change materially.

3—Most of the major fields of the country are now producing a greater volume of high-sulfur crude oil than at any previous time, both in terms of millions of barrels produced and as a percentage of the nation's total oil.

The authors presented extensive charts showing the quantity and percentage of sulfur-containing crudes produced in nine major production areas of the U. S. every fifth year since 1915.

In 1915, according to their figures, only 34% of the crude produced was classed as high-sulfur—over 0.5% sulfur; last year, however, the production of high-sulfur crude had risen to 42% of total crude production. An even higher percentage of high-sulfur crude was produced both in 1925 (44%) and in 1930 (43%), but the quantity of such crude produced was considerably less, of course, since our total crude production today is about double what it was in that earlier period.

The industry-wide seriousness of the sour crude processing problem today was emphasized by the authors in one point they brought out strongly, i.e. that production in 1925 and 1930, although it had almost the same percentage of high-sulfur crudes as of today, was confined largely to the California area, except for the Smackover (Ark.) production. "For this reason," they continued, "it did not affect mid-continent and eastern refiners, but today's production of high-sulfur crudes comes not only from California, but from the West Texas, Mid Continent, Southeastern and Rocky Mountain areas."

Hope Shines Feebly

One ray of hope did shine through, however, when the authors made a comparison of 1946 crude production with estimated reserves. They presented one chart showing 1946 production according to the percentages of various sulfur groups, and a second chart showing estimated crude

API ARTICLES
In this issue

In PETROLEUM PROCESSING this month you will find three articles covering the most important papers presented at the recent meeting of the American Petroleum Institute:

"Where Does the Sulfur Go?"—page 935—the complete text of a paper describing what happens to the sulfur in sour crude when it is processed into finished products.

"Sour Crudes — Equipment Protection"—page 967—the major portions of a paper telling how one refinery protects its equipment from the corrosive effects of sour crude.

"Tests on Cracking Catalysts Used in Control of Commercial Units"—page 945—a staff-written review of each of 14 papers presented at a symposium on the testing and evaluation of cracking catalysts.

oil reserves as of Jan. 1, 1947, also according to the same sulfur groups.

"The most striking thing about the reserves chart," they concluded, "is that it almost exactly duplicates the production chart for 1946. This would certainly seem to indicate that crude oil is being produced in almost all of the fields at a constant ratio to the reserves available in these fields. The significant feature of this fact is that, as long as crude-oil demand requires full production from all fields, no change can be expected in the quantity of high-sulfur crude oil that will be produced.

"The only factors that could make a change would be: (1) a decrease in demand that would decrease the importance of the high-sulfur oil; or (2) discovery of new fields. These fields might yield low-sulfur oil which, of course, would increase its proportion; but, on the contrary, the new fields may have high-sulfur oil, and the ratio of high- to low-sulfur crude oils would become even greater than at present."

Data on what happens to the sulfur when a sour crude is processed was presented in the second paper at the session—"Where Does the Sulfur Go?" by M. J. Fowle and R. D. Bent, Atlantic Refining Co., Philadelphia. The complete text of this paper is published in the current issue of PETROLEUM PROCESSING, beginning on page 935.

In their paper, the authors discussed the distribution of sulfur in products when processing sour crude by various methods: distillation, viscosity breaking, reforming, thermal cracking and catalytic cracking. They also covered treating operations, including mercaptan extraction, acid treating, and catalytic desulfurization.

How to Protect Equipment

The third and final paper on the program—"Sour Crudes— Equipment Protection" by E. Q. Camp, Humble Oil and Refining Co., Baytown—discussed some of the major corrosion problems experienced by Humble in processing sour crudes and the methods used to overcome them. The principal portion of Mr. Camp's paper is also published in this issue, page 967.

Corrosion, according to Mr. Camp, is one of the major problems confronting the designer and operator of refinery equipment, with losses, according to one estimate, being equivalent to approximately 1 cent per gallon of gasoline produced. In addition to the usual corrosion problems experienced by most industries, Mr. Camp continued, the refining industry has some all its own—primarily those which arise as a result of the presence of corrosive sulfur compounds and of chlorides of magnesium and calcium in the crude oils. The major offenders in this respect, he pointed out, are the sour crudes, which are being processed today in increasing quantities.

His paper covered both low-temperature corrosion (below about 500° F.) and high-temperature corrosion (above about 500° F.), and the protective measures employed by Humble for tanks, lines, distillation equipment and cracking units.

Burton Acclaimed Father of Modern Refining Technology

FOR developing with his associates the first commercially successful cracking process, Dr. William M. Burton, the father of modern petroleum refining technology, has been awarded the American Petroleum Institute Gold Medal for Distinguished Achievement.

Presentation of the medal and citation was made Nov. 12 before several thousand representative of all branches of the industry attending the API's 27th annual meeting in Chicago. It was the institute's second such award, the first having been given last year to the late Henry Ford for his work in developing mass-production techniques.

Dr. William M. Burton (left) with the API Gold Medal for Distinguished Achievement and Citation, just presented to him by William R. Boyd, Jr., institute president

Dr. Burton was born in Cleveland Nov. 17, 1865, and received his AB degree from Western Reserve University in 1886 and his PhD degree from Johns Hopkins in 1889. In 1890 he joined Standard Oil Co. as its first chemist at the Whiting, Ind., refinery. He became general superintendent in 1895, a member of the board of directors in 1911, vice-president in 1915, and president in 1918. He resigned the presidency in 1927, but continued in an advisory capacity as a member of the board until 1928. He is at present a resident of Cambridge, Mass.

About 1909, with Dr. Robert E. Humphreys, who had come to Whiting from Johns Hopkins in 1900, and Dr. F. M. Rogers, who joined the staff in 1908. Dr. Burton started the first work ever done in an industrial laboratory toward producing petroleum products by chemical change—work which eventually resulted in the thermal cracking process.

Many difficulties were encountered in getting the process going. Equipment was poor and knowledge slim. The pressures and temperatures were exceedingly high for the metals then available, and even the 100-gallon pilot plant still had to be caulked because it leaked at the rivets and seams when hot.

When Dr. Burton requested $1,000,000 from the board of directors of the then parent Standard Oil Co. to build a battery of production stills, he was told "No, you'd blow the whole state of Indiana into Lake Michigan!"

When Standard of Indiana became an independent unit, Dr. Burton tried again—this time for $800,000. Asked, "are you sure you know what you're doing?" he replied simply "Yes!" and got the money. Events have since proved that he did know what he was doing.

Synthesis of Hydrocarbons from Gas As Projected for Stanolind Plant

A Condensation of a Paper
By GEORGE ROBERTS, Jr., Stanolind Gas & Oil Co.

First description of some of the details of operation and anticipated operating data have been projected for one of the two synthetic-fuels-from-natural-gas plants in this country. Plant is that of Stanolind Oil & Gas Co. in the Hugoton Field in Kansas and was described in a paper entitled "Synthetic Liquid Fuels and Chemicals from Natural Gas" delivered by George Roberts, Jr., of Stanolind before the meeting of the Independent Natural Gas Assn. of America in Oklahoma City, Oct. 24.

Although the effect of processing variables in commercial operation of such plants is not yet known, it is stated that maximum yields of hydrocarbons are obtained in the temperature range of 550 to 650° F. and pressure range of 250 to 500 psi. in the fluidized catalytic reaction step. The limits of space velocities on yield have not been fully determined.

This article is a condensation of Mr. Roberts' paper.

TWO commercial plants for the manufacture of synthetic hydrocarbons from natural gas by the American-improved version of the Fischer-Tropsch process are at present being designed and engineered in this country.

One is by the Stanolind Oil & Gas Co., probably to be built in the Hugoton Field of Kansas, and the other is by the Carthage Hydrocol Corp., whose plant is to be built at Brownsville, Texas.

The plant at Hugoton is to be 100% Stanolind owned and operated. It will have a nominal rating of 6000 b/d of synthetic liquid hydrocarbons of which about 80% is premium-grade gasoline. In addition to the hydrocarbon product, approximately 320,000 lbs./day of oxygenated chemical compounds are to be recovered. A very brief description of the general Hugoton plant design follows.

Oxygen is supplied from two identical plants having a combined nominal capacity of 53 million cu. ft./day of oxygen. Approximately 109 million cu. ft./day of natural gas containing 22% nitrogen are supplied to the gas generator. The reaction temperature is in excess of 2400° F., and almost complete conversion of the natural gas and hydrogen occurs. Incidentally, the operating pressure is about 300 psi. No catalyst is used in this first step.

The product gases from the reaction zone are cooled to 600° F. in a waste heat recovery system in the gas generator. This heat is utilized to generate steam for plant use. Gases leaving the generator are further cooled by exchangers before entering a scrubber tower where any carbon formed in the generator is removed by water.

Part of the gas from the scrubber is heated to about 700° F., steam is added, and the mixture is passed over a catalyst to form hydrogen and carbon dioxide. The purpose of this operation is to control the ratio of hydrogen to carbon monoxide in the fresh feed synthesis gas. This product is then cooled and combined with the main gas stream and sent into the reaction system.

Fresh feed gas from the gas generation plant is mixed with recycle gas from the product separator and introduced into the bottom of the reactor. The catalyst is iron promoted with alkali and prepared by grinding iron oxide to about 100 mesh, adding the promoter, and reducing the iron oxide to the metal with hydrogen. The catalyst is "fluidized" by the upward flowing gas, and the heat of reaction is tran-

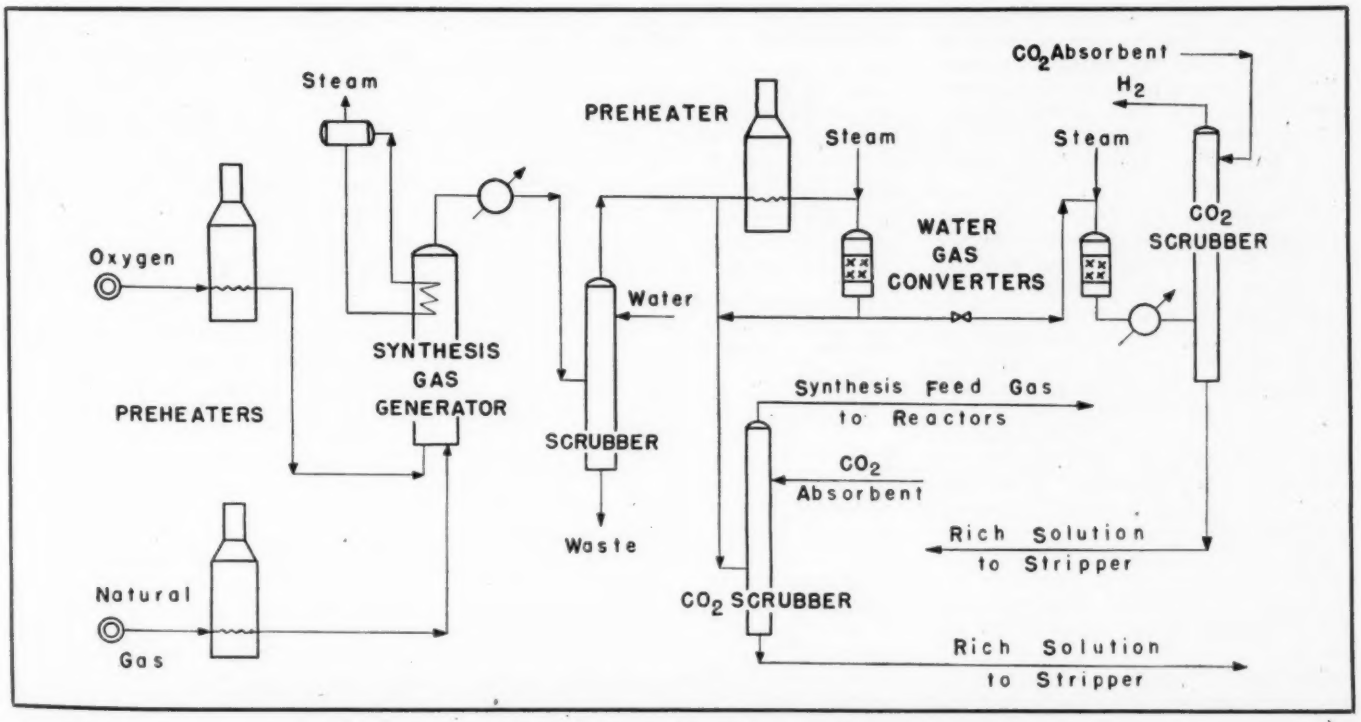


Fig. 1—General flow diagram for synthesis feed gas generation

ferred to internal cooling tubes in which steam is generated at a pressure of 675 psi.

Two reactors capable of operating between 250 and 500 psi. process a combined feed gas of almost one billion standard cu. ft./day. The steam generated in cooling the reactors amounts to about 350,000 lbs./hr. The reactor effluent at 600 to 650° F. passes through heat exchangers and coolers to the primary product separator.

From this separator one gas stream and two liquid streams are withdrawn. A portion of the gas stream is recycled to the reactor while the net product gas enters the product recovery section of the plant. The hydrocarbon stream, containing oil-soluble chemicals, may be processed for recovery of the chemicals or passed directly to refining facilities. The water stream, containing water-soluble chemicals, is processed for recovery of the oxygenated compounds.

Since a large portion of the hydrocarbon product is in the light olefin range, the net product gas must be stripped of these components to augment the plant yield. Adsorption on silica gel or carbon, acid absorption of the olefins, and conventional oil absorption were all considered for this service. Conventional oil absorption was finally selected. The recovered propylenes and butylenes are polymerized over solid phosphoric acid to yield approximately 1000 b/d of polymer gasoline.

The C_5 + liquid hydrocarbons from the primary separator and gas recovery system are processed in a fluid isoforming operation utilizing commercial type catalyst to produce gasoline and heavier fuels. Oxygenated compounds charged to this operation are converted to the corresponding olefins, and, if the oil-soluble chemicals are not removed in prior processing, the yield of gasoline is augmented.

Hydrocarbon synthesis over iron catalyst is possible to some degree at any temperature above 500° F. Maximum yields of condensable hydrocarbons with a given type catalyst are obtained in the range of 550 to 650° F. Higher temperatures produce large quantities of methane and other light hydrocarbons.

The exact effect of variations in pressure is not thoroughly known. At extremely high pressure, chemicals were synthesized by the Germans, and hydrocarbons were obtained as contaminants; however, since the project now planned is primarily interested in synthetic hydrocarbons, it will operate in the range of 250 to 500 psi.

The yield of useful products from the synthesis can be controlled to some extent by variations in the reactor feed gas. The limits of space velocities in this synthesis have not been fully determined.

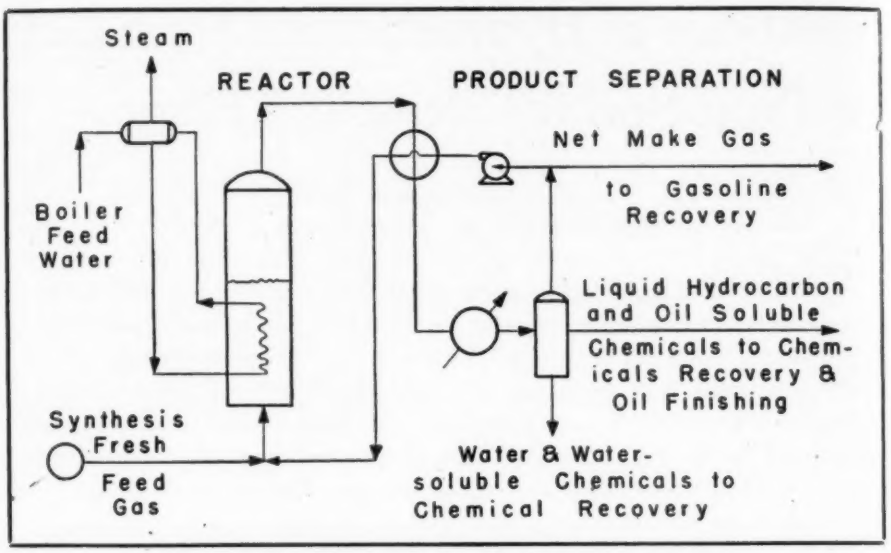


Fig. 2—Flow diagram for synthesis reaction section

The hydrocarbons produced in the synthesis consist principally of straight chain compounds all the way from methane to solid waxes. The major portion of the product is unsaturated in structure with small amounts of corresponding saturated paraffins. This is in sharp contrast to products obtained with the German cobalt catalyst wherein the paraffins predominated. The practical effect of this is that unsaturated products have a much higher octane rating than the corresponding paraffin product.

The oxygenated chemicals formed in the synthesis reaction include alcohols, aldehydes, ketones, and acids. In a plant producing 6000 bbls./stream day of synthetic hydrocarbons, the estimated yield of chemicals is about 420,000 lbs./day, of which about 320,000 lbs. are preferentially water soluble and are in the water condensed from the reactor product. The heavier chemicals are preferentially soluble in oil and are obtained with the liquid hydrocarbon stream from the product separator.

Following are the hydrocarbon products to be made from the Hugoton type of operation:

Product	b/d
Gasoline	5400
Distillate fuel	700
Fuel	300
Total	6400

The finished gasoline product has a clear A.S.T.M. octane number of approximately 80, and the heavier products compare favorably with similar refined products from petroleum. The water-soluble chemicals obtained from the primary separation constitute an appreciable portion of the yield of useful product. Some of the compounds identified in the crude mixture are as follows:

Alcohols—methanol, ethanol, propanol, butanol, and pentanol.

Aldehydes—acetaldehyde, propanal, butanal.

Ketones — acetone, methyl-propyl, and methyl-butyl ketone.

Acids—acetic, propionic, and butyric.

These chemicals occur in quite a complex mixture, and have presented a challenging problem to research people to find ways and means of separating and purifying the individual components. However, it can probably be definitely stated that this can be done successfully to make specification products.

In addition to the two plants to carry out the primary synthesis process at Hugoton and Brownsville, there will also be a commercial chemicals separation and purification plant at each of these locations. Both of these plants will be owned and operated by Stanolind Gas & Oil Co. A contract has been made with the U. S. Industrial Chemicals Co. to handle the refined chemical products from these two plants.

The economic situation in the U. S. is such that, at the present time and under present conditions, natural gas is the most economic source for the production of synthetic fuels. However, the future situation may be such that coal as a basic raw material for synthetic fuels and chemicals will become economic.

Preliminary attention is already being devoted to the improvement of the engineering and technology involved in the mining and utilization of coal for this process, and further work will be done as economic conditions dictate. It can be definitely stated that technical research and development has already made it possible to give assurance that an adequate supply of liquid fuels will be available from natural gas and coal for many, many generations to come.

Improved Design, Operating Techniques For Girbotol Absorption Processes

By R. M. REED, Chief Chemical Engineer
The Girdler Corp., Gas Processes Division

The solution of operating problems in Girbotol process plants for purifying natural and refinery gases has provided a number of improvements which have become current standard design and operating procedures for the processes.

Solution carryover due to foaming, resulting in loss of gas handling capacity, has been reduced by using small quantities of anti-foam agents such as oleyl alcohol. Sludge, such as iron sulfide or sulfur, has been removed by filtration and periodic cleaning of equipment.

Thiosulfate and organic acid contamination, which reduces the effectiveness of the amine treating solution, has been removed by caustic soda with either batchwise or continuous techniques. Economic considerations have brought variations in the choice of treating solutions to meet corrosion problems; use of corrosion-resistant alloys has also been effective. Decomposition of amines or loss by vaporization have been relatively minor problems and have given little difficulty.

THE Girbotol absorption processes for purifying gases and liquids by the use of amine solutions have found widespread acceptance since the first commercial operation in 1929, which used a triethanolamine solution for the removal of carbon dioxide from helium-bearing natural gas in a helium recovery plant at Thatcher, Col.

At present, the Girbotol gas treating process is the only one being installed on a large scale for the removal of hydrogen sulfide from natural and refinery gases, and it is being extensively used for the separation and recovery of carbon dioxide from gas mixtures. Also numerous installations of the Girbotol liquid treating processes have been made for purifying hydrocarbon liquids, such as propane, butane and natural straight-run and cracked gasolines. A typical flow diagram for a gas treating plant is shown in Fig. 1.

As can be expected with any process that has come into general use, a number of operating problems have arisen in connection with various Girbotol process plants. The solution to these and similar problems has led to many improvements in plant design and operating techniques. The following are typical of the problems that have been encountered.

(1) Solution carryover and foaming.

(2) Fouling of solutions by sludge.

(3) Thiosulfate accumulation in solutions.

(4) Accumulation of organic acids in solutions.

(5) Corrosion of equipment.

(6) Decomposition of solutions.

(7) Losses of amines by vaporization.

(8) Incomplete purification.

Solution carryover from the tops of absorber towers has occurred in several plants. In common with other alkaline solutions, amine solutions will react with organic acids to form salts, many of which have soap-like properties, and will cause the solutions to foam when present in them. Organic acids are present at times in natural and refinery gases, and in such cases will promote foaming in amine solutions. This foaming may seriously decrease the gas handling capacity of the absorber.

Fortunately, however, it has been found that the addition of small quantities of suitable anti-foaming agents to the amine solutions will overcome the foaming tendency of the solutions.

In one case, in an absorber in a Girbotol process plant purifying natural gas with aqueous monoethanolamine solution, solution carried overhead at a gas rate of 14 million cu. ft./day. After about 15 parts per million of oleyl alcohol (ocenol) were added to the solution, the gas rate

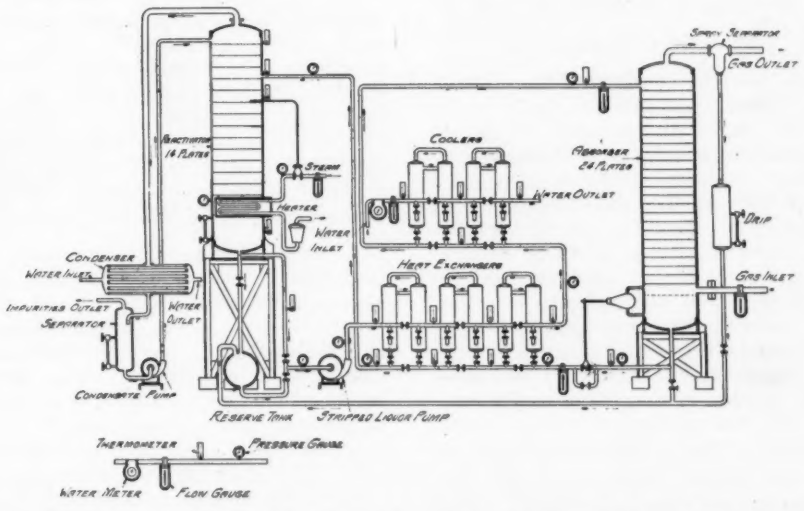


Fig. 1—Flow diagram of the first Girbotol gas purification plant, at the Helium Co. plant in Thatcher, Col. Although several hundred plants for treating gas by this process have been built, flow diagrams of present installations differ in only a few details from that at Thatcher

was increased to 50 million cu. ft./day with no carryover occurring.

Sludge accumulations in amine solutions have proved troublesome at times. The sludge, ordinarily consisting of iron sulfide and sulfur, is carried into the absorber as a dust with the gas stream. Microscopic examination of sludge taken from an amine solution has shown it to be identical with material present in core samples taken from the gas producing formation.

Iron sulfide sludge will also be present in amine solutions in cases in which corrosion of pipelines and equipment is occurring with the formation of iron sulfide. When present in sufficient quantity, iron sulfide sludge may foul heat exchanger surfaces, erode metallic surfaces at points of high solution velocity and plug bubble tray and packed towers.

Various Methods Used

Various methods have been used for overcoming sludge problems, such as the installation of filters to separate sludge from the amine solution, the periodic cleaning of equipment with water and with inhibited hydrochloric acid, and the use of water scrubbers on the gas stream before it enters the absorber. Centrifuges also have been used for cleaning the solutions, and in some cases solutions have been freed of sludge by decantation.

Iron sulfide is not readily wetted by hydrocarbons, so that the dust does not settle out of the gas stream in condensate separators, but carries on into the absorber, where the amine solution scrubs it out of the gas. Although the sludge accumulation occurs at a rate of only a few pounds per day, it frequently becomes troublesome in natural gas purification plants unless some of the above-mentioned procedures are employed for removing it from the system.

Thiosulfate Formation

Another source of contamination of the amine solutions has been in the formation of thiosulfate. This occurs when solutions containing sulfides are contacted with oxygen, present either in the gas stream or by exposure of the solutions to air. The most common case in which thiosulfate accumulation occurs in the amine solution is in plants in which gas collected in vacuum gathering systems is being purified. This gas will frequently contain as much as 0.5% oxygen, present due to air leakage into the system.

Oxygen is also sometimes present in some refinery gases resulting from catalytic cracking operations. When both hydrogen sulfide and oxygen contact an alkaline solution, such as a sodium carbonate or an amine solution, oxidation of the sulfide occurs, resulting in the formation of thiosulfate.

Thiosulfates are relatively stable salts, and amine thiosulfate does not dissociate on heating like amine sulfide or carbonate, so that the presence of amine thiosulfate in the solution decreases its effective amine concentration by a corresponding amount. By adding caustic soda or soda ash to an amine solution containing amine thiosulfate, sodium thiosulfate will be formed and the combined amine will be liberated. In this way the effective amine concentration of the solution may be maintained, until the solution becomes saturated with sodium thiosulfate.

It will then become necessary to separate sodium thiosulfate from the amine solution. This may be done either by steam distillation, stripping the amine out of the solution, or by extraction, separating the amine from the solution by a solvent in which the sodium thiosulfate is insoluble. The operation can be carried out either batchwise, to reclaim a complete charge of solution at one time, or it may be carried out as a continuous operation on a small side stream of the amine solution.

In addition to the traces of organic acids present in gas streams that sometimes cause foaming in amine solutions, there have been a few cases, particularly with refinery gases, in which the gas streams contained sufficient quantities of organic acids (principally formic and acetic acids) so that a fairly rapid accumulation of these acids occurred in the amine solutions. In such cases, a treatment similar to that used with thiosulfate may be employed, namely to add soda to the solution until the sodium salts become concentrated, and then recover the amine from the solution either by steam distillation, or by extraction.

Process Corrosion

Corrosion of equipment has been a serious problem in some Girbotol process plants, while in other such plants little difficulty has been experienced. These plants normally are built entirely of iron and steel, since amine solutions, like ammonia, will dissolve copper and brass and most alloys containing zinc or copper. Aluminum has been utilized to some extent, although it is susceptible to attack in the presence of iron sulfide.

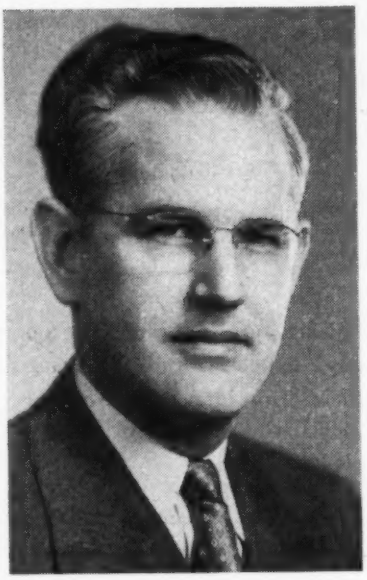
Absorbers usually are free from corrosion difficulties, although two cases have been reported recently in which corrosion cracks appeared near welds in absorber shells and piping after about two years of service. Numerous other absorbers, however, have given satisfactory service for long periods of time, and no particular difficulty is anticipated in avoiding a recurrence of this type of corrosion.

Iron Sulfide One Cause

Rich solution piping and control valves occasionally are subject to corrosion, particularly in cases in which erosion also occurs due to the abrasive action of iron sulfide sludge in the solution. Control valves are normally trimmed with stainless steel, but carbon steel pipe generally has a satisfactory service life. Heat exchangers should be provided with suitable impingement baffles to prevent erosion. Corrosion of carbon steel exchanger tubes on the rich solution side has occurred at times, but the frequency of occurrence of such corrosion has not been great enough to warrant the general use of alloy tubes in solution heat exchangers.

Reactivators have been subject to corrosion in a few plants. In one plant corrosion occurred to such an extent that the reactivator was replaced after 5 years' service, and in another plant the reactivator shell was protected by an outer lining after three years' service. In both plants the natural gas being purified contains more CO_2 than H_2S, and it is felt that the corrosion has been accelerated by the presence of the CO_2.

The Author



R. M. Reed obtained his Ph. D. degree from the University of Washington in 1935 and was associated with Procter & Gamble, Cincinnati, from 1930 to 1937. He joined the Girdler Corp., of which he is cnief chemical engineer of the Gas Processes Division, in 1938. He holds various patents in the fields of gas purification and hydrogen manufacturing.

These plants both circulate aqueous monoethanolamine solutions.

In another plant, heat exchanger and reactivator corrosion occurred at a relatively high rate with a circulating solution that was a glycol-amine solution being utilized for concurrent H_2S removal and dehydration of natural gas, also containing more CO_2 than H_2S. In this case, the corrosion was stopped entirely by replacing the glycol-amine solution with an aqueous amine solution.

This change was made after laboratory studies showed the aqueous amine solution to be less corrosive than the glycol-amine solution. Operating results with the aqueous amine solution verified the laboratory findings. Dehydration of the gas is now being effected with a separate dehydrating solution. No corrosion has been reported in reactivators above the solution inlet or in the outlet pipe to the H_2S cooler even though the concentrations of H_2S and CO_2 are high. H_2S coolers have not corroded to any extent except in cases in which iron sulfide sludge has accumulated on the tubes, or water (saturated with H_2S and CO_2) has lain in contact with them.

Reboilers have corroded in the same plants in which severe reactivator corrosion has occurred, but in most plants no serious difficulty has been encountered. Substantially no corrosion has been experienced in solution pumps, except for the abrasive effect of iron sulfide. No corrosion difficulties have been encountered in solution coolers.

Steam Corrosion

In addition to corrosion on the process side in Girbotol process plants, as described above, there have been other cases of corrosion reported due to corrosive steam or cooling water, which corroded the carbon steel tubes of the equipment in which it was used. The process corrosion experienced in H_2S removal plants has not been associated ordinarily with decomposition of the amine solutions, but has appeared to be due to specific conditions of temperature and concentrations of H_2S, CO_2 and amine occurring in certain parts of the equipment.

As a result of efforts to overcome corrosion in this type of plant, it has been found that some remedial measures are available. For example, use of corrosion resistant materials and changing operating conditions to avoid corrosion have given reasonable freedom from corrosion in several cases.

Decomposition of amine solutions has occurred in a few cases. For example, the monoethanolamine in one plant solution was completely converted to diethanolurea by reaction with carbonyl sulfide present in traces in the gas being purified. In another case monoethanolamine was converted into N-ethanolacetamide by reaction with ketene. Neither of these compounds is alkaline, and neither is convertible back to monoethanolamine on boiling in an aqueous solution.

However, this problem can be overcome either by utilizing amines which do not react with such compounds, or by installing suitable equipment for regenerating the amine from these compounds. There has been no evidence thus far to indicate any thermal decomposition of monoethanolamine in aqueous solutions, even at temperatures as high as 300° F. Thermal decomposition has been noted in a few cases with glycol-amine solutions, presumably due to localized overheating.

Amine Loss Not Serious

Losses of amine by vaporization into the gas stream have not been a serious problem in plant operation, since the amines are relatively nonvolatile, and the small quantity of amine that does vaporize can be readily recovered by a simple water washing section in the top of the absorber. A similar water washing operation is also carried out in the reactivator to prevent amine being lost with the acid gases leaving the plant.

Incomplete purification has been a problem only in plants in which other operating difficulties have been encountered, such as solution foaming or carryover, or in plants in which the necessary provisions have not been made for obtaining sufficient contact between gas and solution in the absorber or between solution and stripping steam in the reactivator.

Improvements in Girbotol Plants

The first Girbotol process plants employed an aqueous solution of triethanolamine as the reagent, and this later was replaced with monethanolamine. This change was made in order to obtain more complete removal of H_2S from the gas than was obtainable with triethanolamine in ordinary absorbers. Monoethanolamine is a much stronger base than triethanolamine (the dissociation constant of monoethanolamine is 5×10^{-5} at 20° C, as compared to 6×10^{-6} for diethanolamine and 3×10^{-7} for triethanolamine), and consequently will absorb acid gases more readily than triethanolamine.

Combination of an aqueous monoethanolamine solution and diethylene glycol has been employed to effect simultaneous desulfurization and dehydration of natural gas with the same solution. In the course of early investigations of such amine-glycol solutions, it was noted that the mixture dehydrated gas more completely than diethylene glycol alone with the same water content, and this was attributed to the hydration of the amine.

However, the hygroscopic nature of the ethanolamines themselves apparently was not fully appreciated at that time. It now is known that monoethanolamine is considerably more hygroscopic than diethylene glycol, while diethanolamine is about equal to diethylene glycol in hygroscopicity.

A plant installed in 1939 employed an 85% solution of diethanolamine to effect simultaneous desulfurization and dehydration of natural gas. The dehydration effected was equal to that obtained with diethylene glycol, and in addition, hydrogen sulfide removal was obtained.

However, the relatively high viscosity of the concentrated diethanolamine solution limited its effectiveness somewhat, and in later plants a solution containing monethanolamine, diethanolamine, diethylene glycol and water has been utilized for the simultaneous dehydration and desulfurization of natural gas. The viscosity of this solution is only about one-fourth that of the concentrated diethanolamine solution, so that it can be processed in heat exchange equipment more effectively than the more viscous solution.

Separate Gas Dehydration

Although considerable publicity has been given to the use of amine-glycol mixtures for the simultaneous desulfurization and dehydration of natural gas, these mixtures are not usually employed for the treatment of gases containing appreciable quantities of H_2S and CO_2. It is general practice to dehydrate such gases separately after purification has been accomplished with an aqueous amine solution.

A modification of the basic Girbotol process cycle, designed to increase the dehydrating power of the absorbent solution, has been proposed by George Sowards of The Ohio Oil Co. and has been placed in operation in the plant of Billings Gas Co. at Garland, Wyo.

In this modified cycle a small portion of the reactivated solution is passed as a side stream through a second reboiler in which substantially all of the water is boiled out of this portion of solution. The dehydrated solution is then cooled and pumped into the top of the absorber tower above the point at which the main solution stream enters. The main solution stream removes the hydrogen sulfide and the bulk of the water vapor from the gas stream, while the comparatively small stream of dehydrated solution serves to dehydrate the gas stream to a very low dewpoint.

The steam requirements are less than if the entire solution were dehydrated to the same extent as the small side stream, and better heat

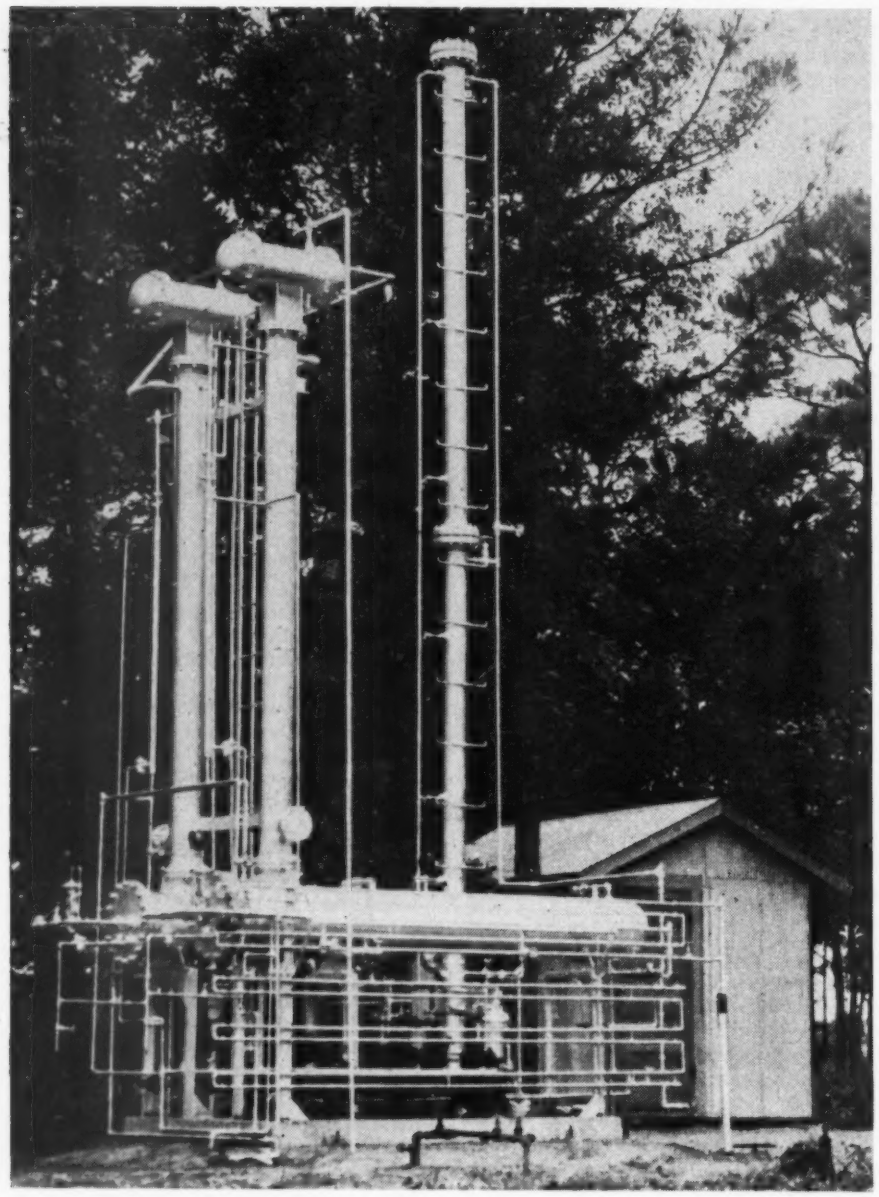


Fig. 2—Girbotol pilot plant in the McKamie field, Ark., installed in 1941. Two reactivators are at the left; the tall slim tower at the right is the absorber

exchanger performance is obtained. With the modified cycle in the Billings Gas Co. plant natural gas is purified to a negative lead acetate test and the dewpoint of the gas is lowered 60-65° F. below the treating tempeature.

Solution heat exchange equipment has been widely used in Girbotol process plants to effect heat economies. However analytical studies of the operating conditions in absorbers and reactivators have shown that, under certain conditions optimum heat recovery can be obtained between rich solution and acid gases rather than between rich and lean solutions. Thus a preheating section may be used in the reactivator to replace conventional solution heat exchange equipment. In cases where this type of operation is applicable, an appreciable saving in equipment cost may be effected with no increase in utility requirements.

H_2S as High as 25%

The quantity of hydrogen sulfide present in natural gas varies over wide limits. Much gas is entirely free from hydrogen sulfide, and large quantities contain only traces. Gas has been discovered recently, however, with hydrogen sulfide contents of over 25%. The purification of these high sulfur content gases imposes special problems in the design of plants for the employment of the process, and also presents the problem of the disposal or utilization of large quantities of hydrogen sulfide.

Solutions for these problems were worked out on a large scale in connection with a Girbotol process plant which was installed in Arkansas for the McKamie Gas Cleaning Co. to purify 30 million cu. ft./day of natural gas containing 7% hydrogen sulfide and 5% carbon dioxide. The hydrogen sulfide recovered in this plant is sold to the Southern Acid and Sulphur Co., which converts it to elemental sulfur by controlled oxidation with air. From 60 to 75 tons/day of elemental sulfur are produced from hydrogen sulfide in the sulfur recovery plant.

At the time the McKamie gas field was discovered, no gas with this high a hydrogen sulfide content was being purified for pipeline distribution. To study the problems in purifying this gas and to obtain other useful information, the Girdler Corp. erected a pilot plant in the McKamie field and conducted a series of tests on the purification of this gas.

This pilot plant is shown in Fig. 2. It will be noted that the pilot plant contained two reactivators and reboilers, and one absorber. This was done so that tests could be conducted on two-stage reactivation. It was found that by preliminary reactivation of aqueous monoethanolamine solution at atmospheric pressure, followed by a second reactivation at higher pressure (30-45 psig.), H_2S and CO_2 could be recovered separately from the solution.

It was also found that by operating single-stage reactivation at pressures of 30-45 psig. the acid gas capacity of the solution could be increased considerably over the capacities obtainable with atmospheric pressure reactivation.

Tests conducted at the McKamie pilot plant also included comparative studies of aqueous monoethanolamine solutions, as well as amine-glycol solutions. It was later operated for several months to provide hydrogen sulfide for use in a sulfur recovery pilot plant operated by the Texas Gulf Sulphur Co.

Following the McKamie pilot plant operations, a commercial gas treating plant was installed for the McKamie Gas Cleaning Co. which utilizes an aqueous monoethanolamine solution, reactivated at 35 psig. Fig. 3 is a photograph of the commercial plant.

Another adaptation of the gas treating process has been the development of plants for the purification of natural gas in remote locations, where no utilities other than natural gas are available, and where only part-time supervision can be maintained. This has been accomplished by utilizing direct gas heating for reactivating the amine solution, with cooling by air or by heat exchange with the gas stream, and with solution pumping by gas pressure.

In addition to its use in the particu-

lar fields mentioned above, the Girbotol gas treating process is finding widespread use at present in other applications such as the production of pure hydrogen, the recovery of carbon dioxide, the production of inert atmospheres, and the production of synthesis gas mixtures for ammonia, methanol and Fischer-Tropsch syntheses.

Historical

The Girbotol absorption process for treating gases was discovered by R. R. Bottoms of the Girdler Corp. who began studying the problem of carbon dioxide removal from natural gas in connection with helium recovery as early as 1925, and sometime later found that certain amines would react with carbon dioxide to form carbonates that could be decomposed on heating. However, none of the amines commercially available at that time was particularly suitable for use in a cyclic process based on the above reversible reaction.

When triethanolamine became commercially available in 1928, Bottoms tested it for removing carbon dioxide from other gases, and found it suitable for this purpose. An aqueous triethanolamine solution (25 to 50%) was found to absorb carbon dioxide quite rapidly and completely from a gaseous mixture, while the absorbed carbon dioxide was readily expelled from the triethanolamine solution on boiling at atmospheric pressure. Since triethanolamine is relatively non-volatile (b.p. 394° F. at 5 mm. Hg.), practically no amine is lost when an aqueous solution is boiled.

Following laboratory tests with triethanolamine, the Girbotol gas treating process was put into commercial operation in 1929 for the removal of carbon dioxide from helium-bearing natural gas in a helium recovery plant at Thatcher, Col. where The Helium Co., then a subsidiary of Girdler Corp., had a supply of natural gas containing 8% helium and 15% carbon dioxide. The Thatcher plant purified about 300,000 cu. ft./day of natural gas, reducing the carbon dioxide content to less than 1%.

This was accomplished by circulating 12 gals./min. of 30% triethanolamine solution through a bubble tray absorber to contact the gas stream (at 300 psig. and 120° F.), with the solution flowing from the base of the absorber through heat exchangers to a second bubble plate tower (the reactivator) in which the carbon dioxide was stripped from the solution by steam generated by boiling the solution in a reboiler at the base of the reactivator. The reactivator operated at substantially atmospheric pressure (temperature 215° F.) with the steam and carbon dioxide flowing overhead to a condenser, in which the steam was condensed and the condensate returned to the solution.

The carbon dioxide, which was relatively pure, was continuously vented to the atmosphere. The regenerated triethanolamine solution from the reactivator flowed through a heat exchanger and cooler, and was pumped back to the absorber. The temperature of the amine solution increased about 15° F. in passing through the absorber, due to the exothermic heat of the reaction between triethanolamine and carbon dioxide. A flow diagram of the Thatcher plant is shown in Fig. 1.

Although several hundred plants for employment of the Girbotol gas treating process have been built since 1929, the flow diagrams of many present-day plants will differ from that of the Thatcher plant only in a few details, such as the location of the solution pump, and the arrangement of the reserve tank. The Thatcher plant operated successfully for about two years, and was shut down when the helium plant suspended operations.

Following the successful use of triethanolamine solution for removing carbon dioxide from natural gas at Thatcher, studies were begun to find further applications for this amine absorption process. The next commercial application for the Girbotol gas treating process was the removal of hydrogen sulfide from natural gas. Two plants were placed in operation in 1930. Like the Thatcher plant, triethanolamine was used as the reagent at first, but later, monoethanolamine was placed in these plants and they have since been operated with the latter material. One of these plants, that of Shell Petroleum Corp. at Hobbs, N. M., was the first in which the lead acetate paper test was employed for testing the purified gas for the presence of H_2S, a test which has since been widely used for checking the purity

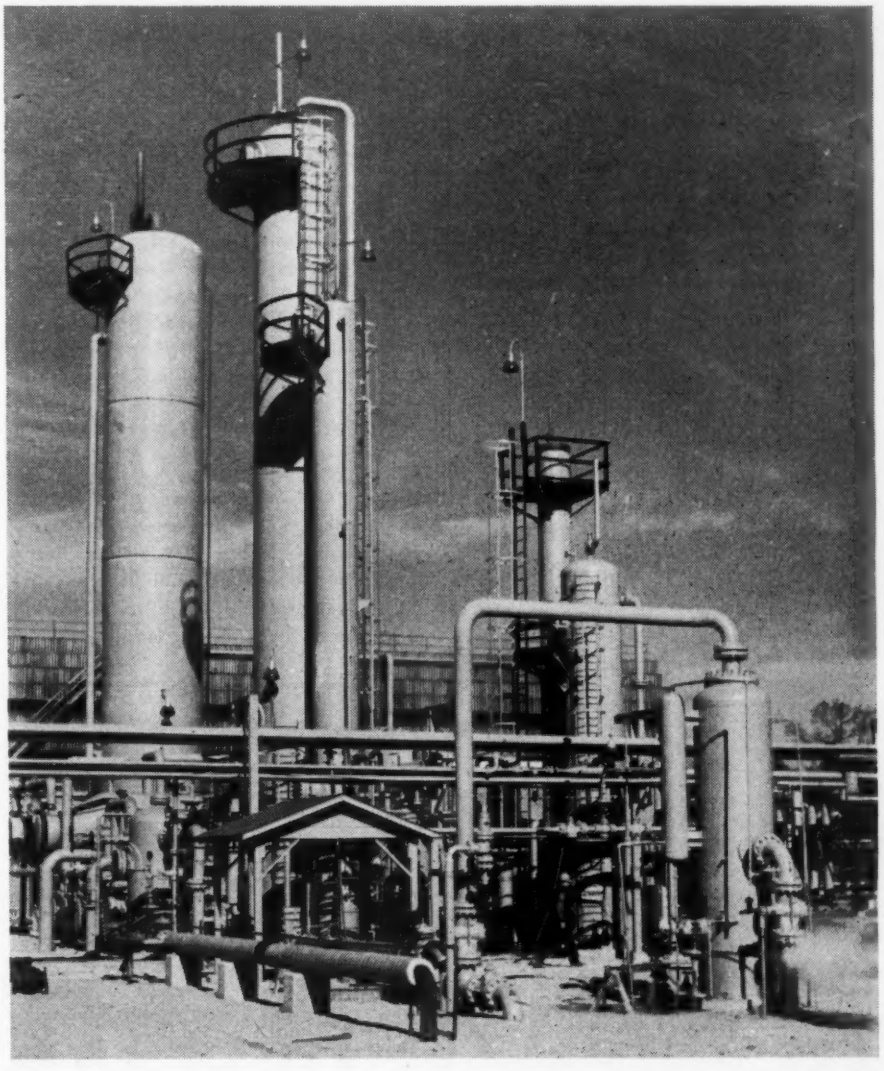


Fig. 3—Commercial gas treating plant installed by Girdler at McKamie, Ark., following work with pilot plant shown in Fig. 2. First column at left is the reactivator; two absorbers in the center; dehydration reactivator and absorber at the right

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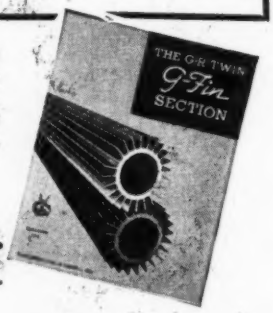

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of purified natural gas for pipeline distribution.

Another application of the Girbotol gas treating process of interest to the petroleum industry is its use for the removal of carbon dioxide from hydrogen produced by the hydrocarbon-steam process. A plant of this type was placed in operation by the Standard Oil Co. of Louisiana at Baton Rouge, La., in 1931. The carbon dioxide content of the hydrogen is reduced from approximately 20% to less than 0.2% in a single absorber. This plant used a triethanolamine solution at first, followed by diaminoisopropanol solution which for a time appeared to have more advantageous properties, after which monoethanolamine solution has been used, since the latter was found to be the most suitable reagent for this particular type of scrubbing operation.

Purification of natural gas by the Girbotol process for pipeline distribution was first practiced commercially in 1937 by the Cities Service Gas Co. at Lyons, Kansas. The Lyons plant employed an aqueous solution of monoethanolamine, and reduced the H_2S content of the gas from 150 grains per 100 cu. ft. to a negative test by lead acetate paper.

Several other Girbotol process natural gas purification plants were installed in Kansas shortly after the Lyons plant. These plants all purified natural gas for pipeline distribution, removing about 50 grains of hydrogen sulfide per 100 cu. ft. from the gas. Since diethylene glycol had been used commercially for dehydrating sweet natural gas somewhat earlier than this, a combination of aqueous monoethanolamine solution and diethylene glycol was tried out in one of these plants to effect desulfurization and dehydration simultaneously. The operation proved successful, and amine-glycol solutions therefore were used also in other plants.

The first application of the Girbotol gas treating process to the removal of hydrogen sulfide from refinery gases was in 1938, at the Atlantic Refining Co. refinery near Port Arthur, Texas. This plant purifies the overhead gas stream from a gasoline stabilizer, reducing its H_2S content from 300 grains of H_2S per 100 cu. ft. to less than 10 grains. The plant capacity is 8,000,000 cu. ft. per day, and an aqueous diethanolamine solution is employed as the reagent.

A Girbotol liquid treating process was applied to the purification of liquid hydrocarbons (gasoline) in 1942, in a plant near Charleston, W. Va., for the Virginian Gasoline and Oil Co. This plant purifies 1000 b/d of natural gasoline, reducing its H_2S content from 50 grains per gal. to 1 grain, utilizing an aqueous diethanolamine solution.

PLANT PRACTICES

Practical Tips and Ideas for Improving Refinery Operations

Oil-filled Valve Trap Mitigates Corrosion On Furfural Solvent Lube Refining Unit

By E. K. DEWEY, Jr., Metallurgist, Manufacturing Department
Continental Oil Co., Ponca City, Okla.

A FREQUENTLY encountered problem with relief valves on processing towers is corrosion of valve seats, discs and guides by certain tower products constantly in contact with them. This results in either sticking of working parts or leaking, neither of which can be tolerated.

Continental Oil Co. has successfully solved this problem at its Ponca City refinery by means of the installation shown in the accompanying photographs and sketch, Fig. 1. In essence, the idea consists of a trap filled with non-corrosive liquid, which prevents direct contact of tower liquid with the valve except when the valve "pops", and a system of drains which keep the valve free of liquid when not in operation.

As installed on the furfural tower of a solvent lube refining unit, the photographs show two views of the valve mounted on the side of the tower.

The trap, or loop, is filled at installation with Conoco's "Dectol medium," equivalent to an SAE-30 oil. If the valve "pops," the oil is replaced by pumping it into the loop through the 1-in. connection at the bottom. This bottom connection also serves to check for and drain off any water which may accumulate.

The initial installation has been so successful that the method has been extended to other trouble spots on the solvent units. Maintenance has been reduced and safety and dependability increased. Conoco's inspection department has conditionally approved the method, which was developed by Mr. J. P. Sinnes, foreman of lube solvent units at Ponca City.

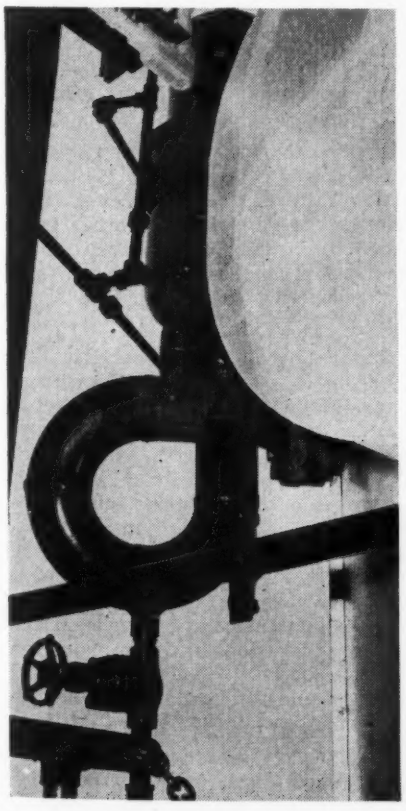
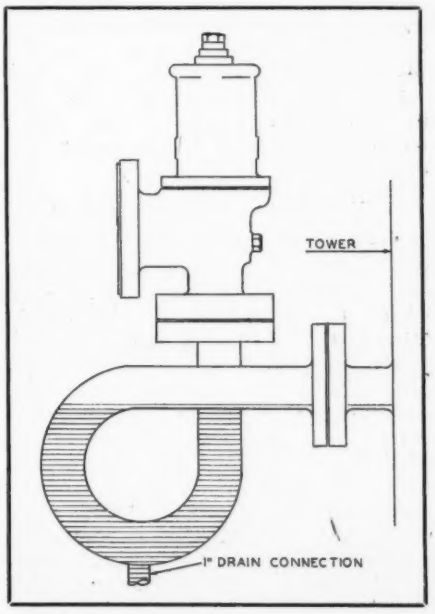


Fig. 1—Below: simplified sketch of trap arrangement on relief valve of furfural tower at Continental Oil's Ponca City refinery. Trap is filled with SAE 30 oil or its equivalent as shown by the shading. At left is a photo of the unit showing connection at bottom of loop for draining and filling trap. At right is another view of the device



Save 83% in Maintenance Labor By Change in Exchanger Bolts

Close working quarters and hot equipment led maintenance engineers at Sun Oil Co.'s Marcus Hook refinery to develop an improved method of bolting end plates on high-pressure heat exchangers, resulting in an 83% saving in labor.

The improvement was in the use of shorter stud bolts with flat-sided, pentagonal heads, as shown in Fig. 2 at B. Previous method, shown at A,

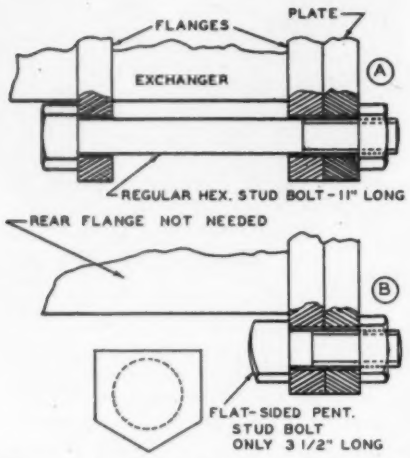


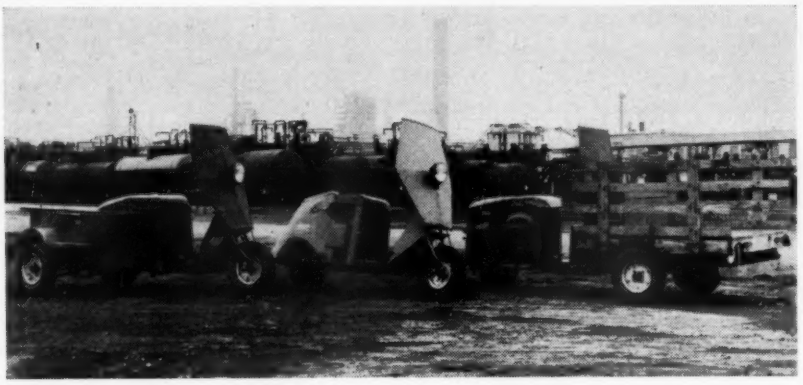
Fig. 2—Improved bolting method for end plates at (B). Previous method using long bolts is at (A)

was use of a regular hexagonal head stud bolt 11 in. long.

With the old method, workmen had to reach between the hot exchanger and its closely adjacent neighbor so as to hold the bolt head with a wrench while he tightened the nut. With the new method, the flat-sided bolthead bears against the exchanger casing, thus eliminating the necessity of holding it with a wrench.

The maintenance job on a bank of high-pressure heat exchangers used to require 3 men working 8 hours for a total of 24 man-hours. Now, 2 men can finish it off in 2 hours.

Use Motor Scooters to Get Around Sprawling Refinery at Wood River



Three types of motor scooters enable Standard Oil Co. (Indiana) personnel to reach quickly widely-scattered locations in the Wood River, Ill., plant. The three-wheeled, side-car type shown at left, is assigned to the control laboratory for sample-takers and to general utility workers. The two-wheelers, seen in the center, are used by operating and maintenance foremen. Particularly valuable are the three-wheeled scooters with truck body, seen at right. Tool room, electric shop, inspection department, and survey crews haul valves, tools, and small but heavy equipment in these. Valve inspectors like them for transporting valves to a central location where specialized test equipment is available, rather than bringing test facilities to the field

"Straddle" Support Among Improvements In Huge Refinery Blowdown Vapor Stack

By JOHN C. ALBRIGHT

TO DESIGN, fabricate, and erect a refinery blowdown vent stack of ample capacity and sufficient height to insure complete safety to personnel and property requires considerable study, particularly when the top of the stack must reach a point some 200 ft. above the concrete foundation.

These, and other problems, confronted the design engineers of Union Oil Co. of California when the old vapor disposal stack and receiving vessel in the Wilmington refinery approached the end of its serviceable life.

The problem of preventing deterioration of the steel walls of the unit from corrosive attack by acid water collected in the receiving vessel entered into the design of the new equipment. The installation is now protected with all facilities economically practicable.

Stack itself is a cylinder, 36 in. outside diameter and about 180 ft. high. Its wall thickness is ½ in. for the lowest 28 ft., with the remainder formed from ⅜ in. stock. Weight, including ladders, guy-line lug rings, and steam lines is approximately 15 tons.

Use Plant Steam System

A steam line is laid from the regular plant steam supply to the unit and extended to the top of the stack where it is connected to a pipe ring. Equipped with 36 nozzles inclined toward the center of the circle, this pipe ring acts as an aspirator and vapor diffuser. Steam flow is automatically controlled for use only when vapors pass through unit and out of stack.

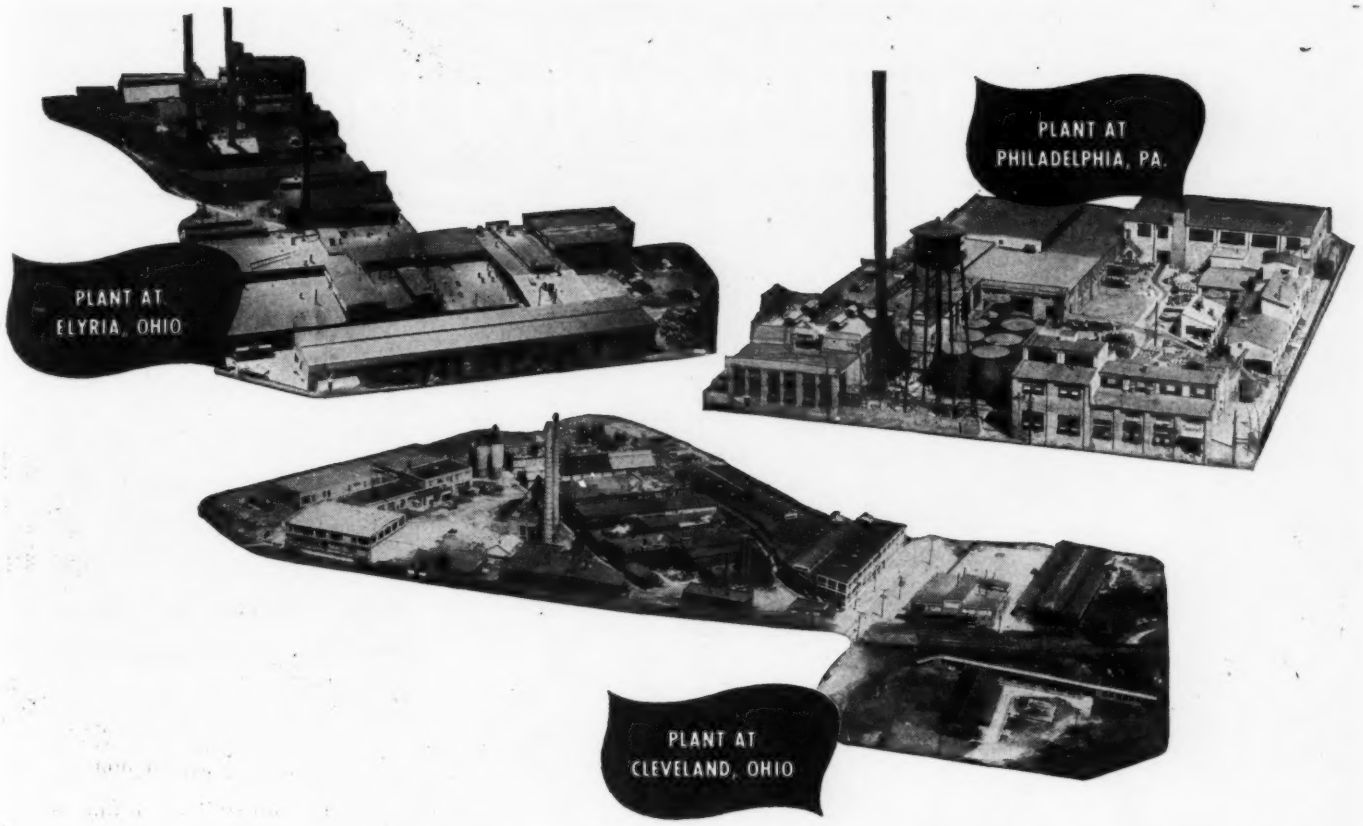
Because of the weight of the stack, it was not considered good engineering practice to mount it directly upon the top outlet of the vapor receiving vessel. Instead, a support of wide flange structural steel was built. "Straddling" the receiver, this structure supports the entire 15 tons of the stack. It is sheathed in concrete as protection against high temperature warping in case of fire.

Two reasons are advanced for use of the straddle type support. One is to eliminate as much stress as possible on the receiver; the other is to provide facilities for replacing the receiver when corrosion conditions warrant without disturbing the stack setting.

The receiving vessel, which disposes of all the released vapors from approximately half the Wilmington refining facilities, is 20 ft. high and 18 ft. outside diameter, all welded construction with a conical roof. It is connected to the stack with ordinary piping of proper diameter. No expansion joint is needed; the cone top absorbs any vertical expansion.

Because the greatest damage in the previous installation was attributed to corrosion and the receiver took the brunt of these conditions, the floor and the walls to a height of 7½ ft. have been covered with 8 lb. sheet lead.

All seams are burned and the sheets of lead are retained against



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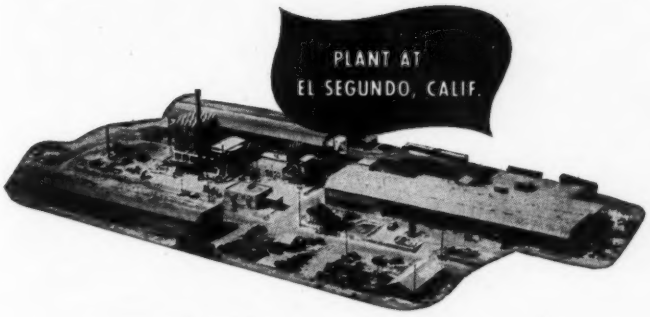

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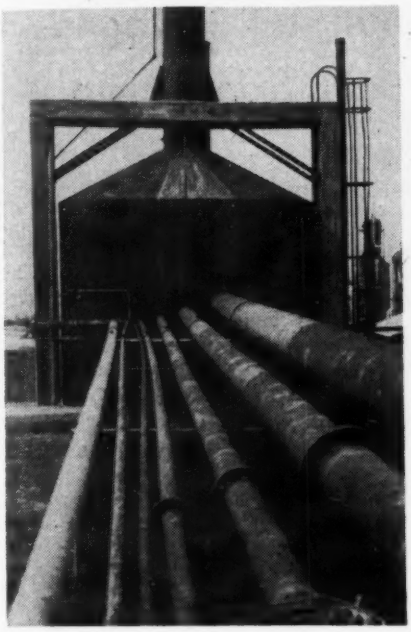

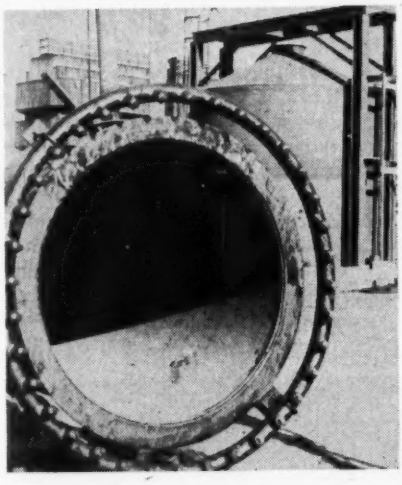

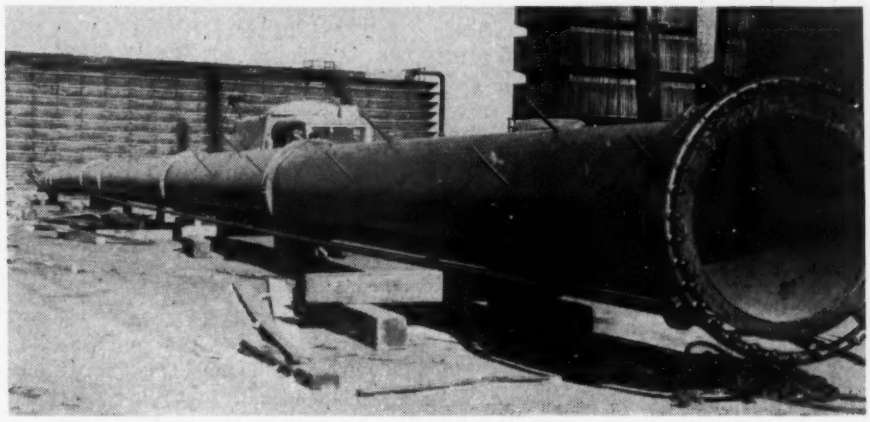

Above: Union Oil's vapor vent stack before installation on top of straddle support. Left top: Stack is in place, straddle support has been covered with concrete insulation; various pipelines carry blowdown vapors from refinery units to receiver. Left center: vapor diffusing steam nozzles in ring at top end of stack. Left bottom: receiver sidewall showing fastening method for inner lining

the walls with square steel washers held snugly against the lead with stove bolts. Each retaining washer, including the bolt head, is covered with a lead patch which also has burned seam construction. The vessel, including lead lining, but exclusive of fittings and connecting piping, weighs about 20½ tons.

Vapor inlet connections are all placed on one side of the receiver, with nozzles entering through flanged connections. Each nozzle serving separate lines is lead lined from the block gate valves to the end inside the vessel.

To prevent undue erosion of the lead lining of the receiver, each nozzle is bent downward at an angle of about 35° so as to spread the vapors more evenly over the floor and opposite sidewall.

Liquids collecting in the receiver—water and oil—are released through liquid level control instruments and drained to the refinery waste pond.

Adapter Cuts Washing Time on Mud-drum

TURBINING tubes in high-pressure boilers usually leaves a deposit which will eventually wash down into the mud-drum, along with sediment from the water.

When this material accumulates, it may carry over into the blowdown valve, plugging it, and necessitate removing the valve to wash out the drum with a water hose. In addition to the time required for unflanging the valve, the connection often leaks

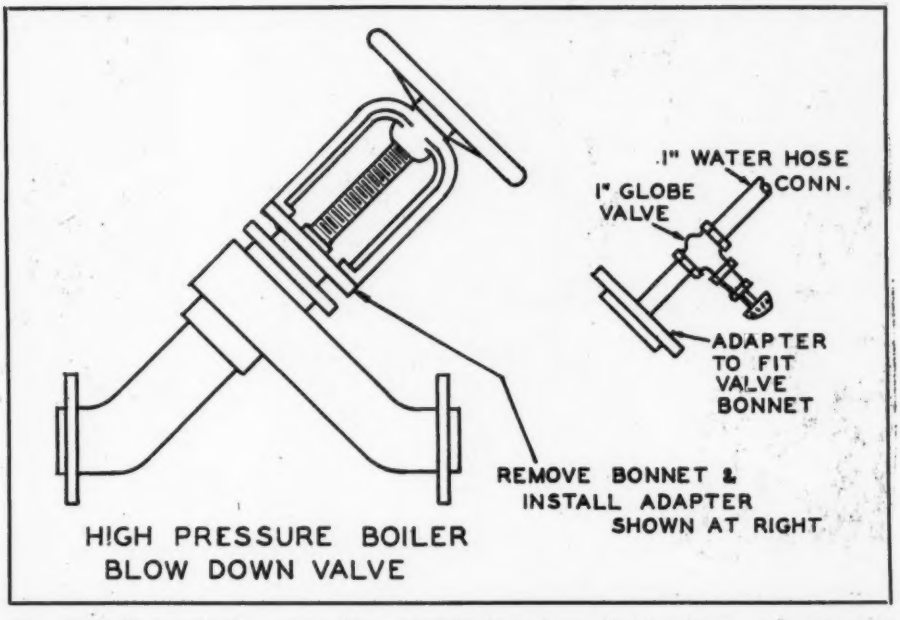
1" WATER HOSE CONN.
1" GLOBE VALVE
ADAPTER TO FIT VALVE BONNET
REMOVE BONNET & INSTALL ADAPTER SHOWN AT RIGHT
HIGH PRESSURE BOILER BLOW DOWN VALVE

Fig. 3—Boiler mud-drum can be water-flushed through blowdown valve by replacing bonnet with special adapter

when the boiler is returned to service, thus requiring an emergency shutdown for repairs.

G. W. Morris, boiler operator for Humble Oil & Refining Co. at Baytown, Texas, has reduced turnaround time for this operation by two hours per job. His suggestion consists of removing the valve bonnet and substituting an adapter which carries a globe valve and a connection for a 1-in. water hose. The drum can be cleaned of accumulation of deposit by alternately flushing and draining.

Fig. 3 illustrates Mr. Morris' idea, which applies particularly to Connelly boilers as shown. It can be fitted to most other makes by appropriate changes in the adapter so as to fit the bonnet of the valve in use.

IDEAS—Wanted!

Plant operators, foremen, superintendents — Send in your own original contributions on "how we do it around our refinery." Possible subjects could include operating shortcuts, money-saving gadgets, safety schemes.

The idea's the thing—so send it in to:

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PETROLEUM PROCESSING
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700 Pipe Flanges per Month Are Salvaged On Beveling Machine Built from Scrap

By VERNON STARR, Welding Shop Foreman
Socony-Vacuum Oil Co., Inc., Lubrite Division, East St. Louis, Ill.

AN ESTIMATED 700 pipe flanges have been salvaged per month during a single daily shift working a regular 40-hr. week in the welding shop of the East St. Louis refinery of Socony-Vacuum Oil Co., Inc., Lubrite Division.

The work is done on a home-made beveling machine built in the shop from scrap steel, discarded bearings, part of an old lathe, and the like. It is used chiefly to bevel weld-type pipe flanges salvaged from dismantled pipe systems, and is shown in the accompanying photographs.

Pipe table consists of a pair of adjustable pipe rolls, belt-driven by an electric motor operating through two gear-type speed reducers so that its speed is approximately one rpm.

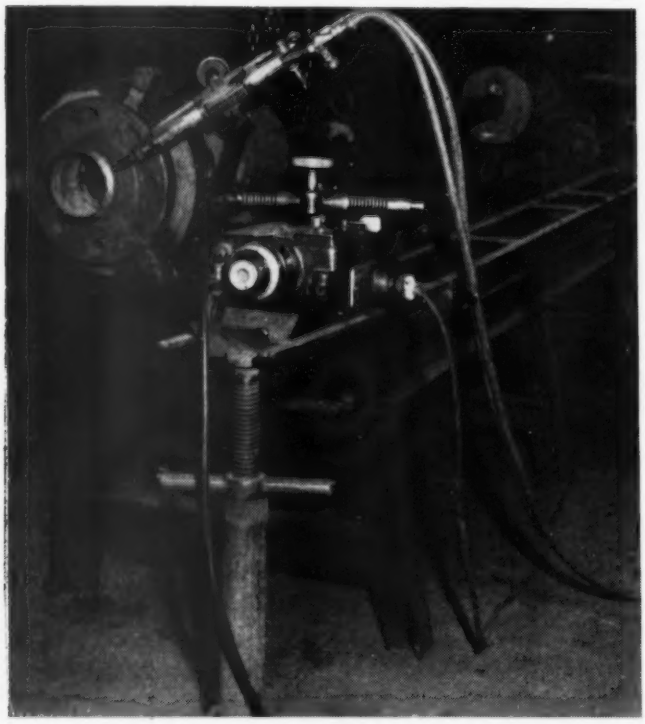
The jig holding the flange is made from an old lathe chuck, mounted on a 2-in. shaft about 6 ft. long and capable of holding a flange up to 14-in. diameter. Welded to the shaft near each end are two rolls, each 14 in. long, made from old 8-in. pipe and supported by discs cut from steel plate.

The flange-holder and flange are placed on the pipe table so that the two 8-in. diameter rolls are in contact with the pairs of driven rolls. The difference in size between the rolls gives the flange a resultant speed of about one-half rpm., sufficiently slow for oxygen cutting operations.

A conventional cutting torch with a 45° cutting tip is mounted in any convenient holder (in this case a regular cutting holder for cutting steel plate) so that the flame impinges on the flange at a 30° angle. Cutting is done in the usual manner.

The salvaged flange has about a 30° bevel and is ready for re-use as soon as the face has been trued. When the cutting starts, attention may be given to other duties until the operation is completed.

The machine shop uses a carboloy-tipped tool for this job. However, the tool is as large as the usual tool holder instead of the small type. With such a massive tool, the later facing cut may be taken with but one setting of the lathe, saving the machinist's time as well as wear on the tool, since the massive tool will not be damaged by the rough surface or by making a very heavy cut.



In photo at left above, a 30° bevel has just been cut on a salvaged pipe flange on the machine built in the welding shop of Socony-Vacuum's Lubrite Division refinery at East St. Louis. In photo at right, large diameter pipe can also be rebeveled on the machine by placing it directly on the driving rolls

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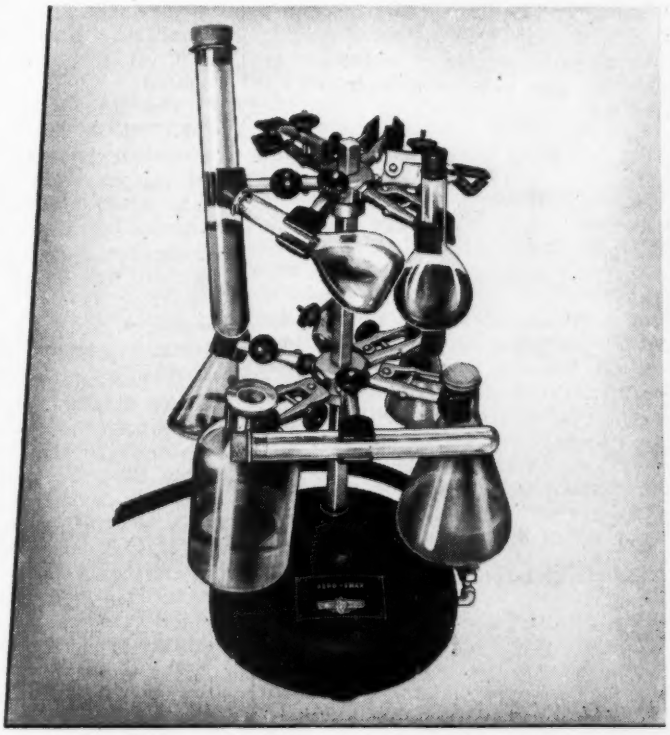

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Improved Design Features Used In Lion's 4500 b/d TCC Unit

LION Oil Co.'s new Thermofor Catalytic Cracking unit, recently placed on stream at the company's refinery at El Dorado, Ark., has already demonstrated its value, according to T. H. Barton, chairman of the board of Lion.

"Designed to process 4500 b/d of fresh gas oil, with 900 b/d additional capacity for recycling, the new TCC unit maintains runs substantially in excess of its rated capacity and exceeds the guaranteed percentage yield of high octane gasoline," Mr. Barton said.

The unit incorporates recent improvements in catalytic cracking, including the use of concurrent flow of catalyst and vapor, recycling to give greater flexibility in production, and alloy steel linings in major items of equipment, he explained.

Process used in the unit was licensed by the Houdry Process Corp., and the engineering and construction was done by the Lummus Co. Total cost of the unit was approximately $1,500,000.

The feed is a heavy gas oil "charging stock" similar to fuel oil and is taken directly from the crude by distillation. Products obtained are gasoline, gas, butanes, burner oils and fuel oil.

Feed Preparation

In the feed preparation section 4700 b/d of the gas oil charge is heated by heat exchange and then mixed with 900 b/d of recycle gas oil separated from the cracked products. This combined stream is then heated until it is principally vapor by three upright heaters, two of which operate in parallel. Any unvaporized tars are removed (about 200 b/d) and the vapors are further heated in a superheater furnace. The final product of the feed preparation section is a stream of 5400 b/d of vaporized heavy oil at 880°F.

Catalytic Section

The catalyst used in the cracking section is the "bead" catalyst, developed during the war in the aviation gasoline program. The three principal pieces of equipment in this section—reactor, kiln, and elevator—are supported on a concrete and steel structure, the highest point of which is 200 ft. above grade.

The reactor, a 12 x 97 ft. vessel, was prefabricated at the Houston shops of the Wyatt Metal and Boiler Works and delivered to El Dorado by special train. The upper section is the storage hopper for the catalyst to the reactor. The central section consists of catalyst storage and a pipe or "leg" for carrying the catalyst downward. The weight of the catalyst in this leg is sufficient to maintain the flow of catalyst against the pressure in the reactor.

The bottom section is the reactor proper where the cracking takes place. In operation the hot burned catalyst from the top of the elevator flows into the storage hopper and then down the leg and into the reactor. The catalyst is distributed over the reactor and flows through it slowly as a bed 12 ft. in diameter and 11 ft. deep.

Hot oil vapors from the feed preparation section enter the reactor above the catalyst level and flow down through the catalyst bed. At the bottom the product vapors are disengaged from the catalyst and flow to the fractionating section to be separated into the final products.

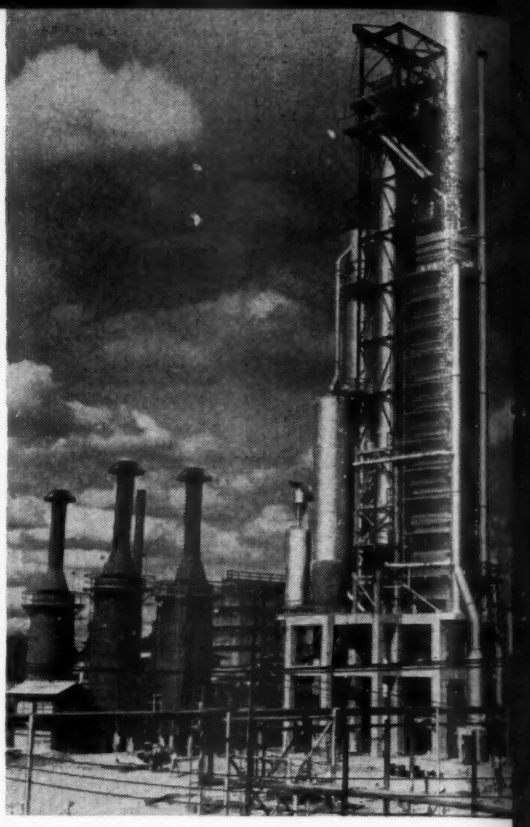
The catalyst, which now contains about 3% carbon, returns to the bottom of the elevator. This downward flow of both the catalyst and oil vapors, known as concurrent flow, is a very recent development and makes possible a lower temperature of the hot oil vapors to the reactor, it is stated.

The kiln is a rectangular vessel, 10.5 ft. by 10 ft. and 95 ft. long. Prefabricated sections were assembled at the site and the entire structure was lifted into place as a unit. The interior lining of refractory, the steam coils, air ducts, etc., were then installed. The kiln is divided into nine "burning zones" each of which is equipped with an air inlet and outlet and a steam coil.

The elevator is a single bucket and chain arranged to carry simultaneously both the coked catalyst from the reactor and the regenerated catalyst from the kiln. The hot catalyst from the kiln is carried in a central section of the bucket and the colder catalyst is carried in sections adjacent to the chain. This protects the chain from the high temperatures of the regenerated catalyst. In wartime units of this type two chains were used.

The elevator is suspended from the top and is driven by a sprocket wheel connected to a steam turbine. It is enclosed in a shaft or tube filled with inert gas. The flow of the catalyst through the reactor and the kiln is controlled by valves at the bottom of the elevator that feed the catalyst to the elevator. This flow is 75 tons/hr. for each stream or 150 tons total.

The low pressure air required for the kiln is supplied by a centrifugal compressor, and an "air-line" burner in the air duct to the kiln furnishes the heat required for start-ups. An "inert gas generator" furnishes a non-oxidizing gas for the elevator shaft and for stripping oil vapors from the catalyst.



Lion Oil's new 4500 b/d TCC unit at El Dorado, Ark. Left to right in structure are reactor, elevator tube, and regeneration kiln. Three upright feed preheaters are at left of unit

An "elutriator" removes disintegrated catalyst, or "fines", by blowing them from the catalyst stream with gas. A steam drum and feedwater preheater are necessary with the steam coils of the kiln. Catalyst storage must be provided for emptying the vessels during shutdown. A number of accurate instruments are required for the precise control of the operation.

Product Separation

Hot reactor effluent vapors from the cracking unit then enter the bottom of the fractionator, a 20-plate column, 12.5 x 64.5 ft., lined with stainless steel for the lower two-thirds as a guard against corrosion.

Overhead vapors from this column consist of the gasoline and lighter gases; this is condensed, cooled, and stabilized in a debutanizer before going to storage. Removed gases are compressed and sent to the refinery gas recovery system where they are separated into fuel gas, butanes, and a fraction that is polymerized into a high octane "poly" gasoline.

Sidestreams from the main fractionator are a high quality burner oil, a circulating stream of hot gas oil which is used to furnish heat for the gasoline stabilizer and for the cold feed to the unit, and 900 b/d of recycle stream. Bottoms consist of a stream of heavy fuel oil, containing the tars formed during the reaction.

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Spent Catalyst Stripper Separate from Reactor In New Fluid "Cracker"

AN IMPORTANT design feature of the new 4000 b/d rated capacity Fluid catalytic cracking unit of The Texas Co. at Casper, Wyo., is the outside spent catalyst stripper pictured at right. This is the first Fluid unit in which the stripper has been built separate from the reactor. Spent catalyst is withdrawn from the reactor through a slide valve which can just be seen to left of midpoint of the stripper.

In the stripper hydrocarbon vapors are separated from the catalyst with steam and returned to the top of the reactor, while the spent catalyst laden with coke falls from the bottom of the stripper and is blown to the regenerator for reactivation. Not only is stripping efficiency increased by this outside arrangement but also all possibility of the stripped vapors being picked up by more spent catalyst, or of "short circuiting" (using spent catalyst over again), is eliminated. Preventing oil from entering the regenerator with the spent catalyst reduces the size of the regenerator and the air blowing equipment required.

Another new design feature of the Fluid unit at Casper is a new cylindrical electrical precipitator designed to operate at high pressure, which replaces the former box type. It saves one-third of the cost of former designs through greater ease of fabrication on a leak-free basis and through reduction of tre rein forcing required.

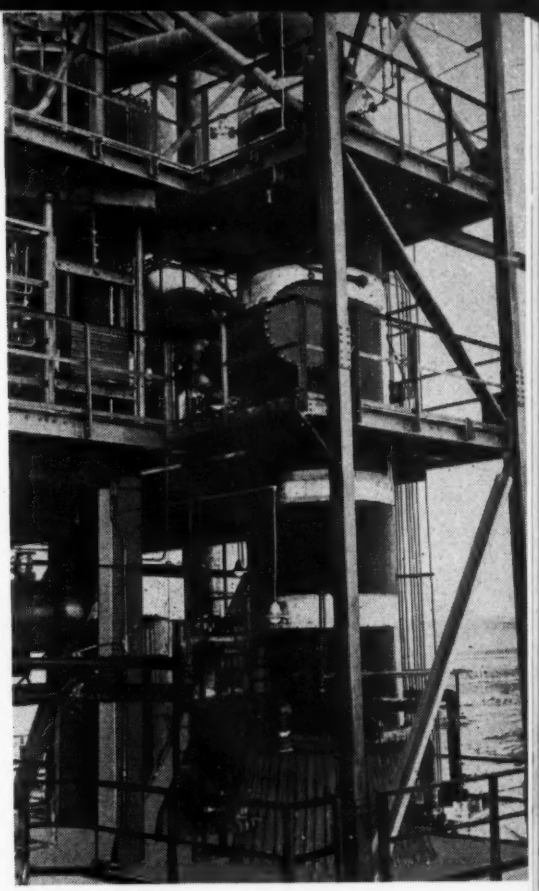
The fractionator of the Casper unit has been consolidated into a tri-functional vessel with a rectifying section, desuperheater and slurry settler included in one tower, which reduced initial cost and improved the safety of the unit by eliminating danger of flash.

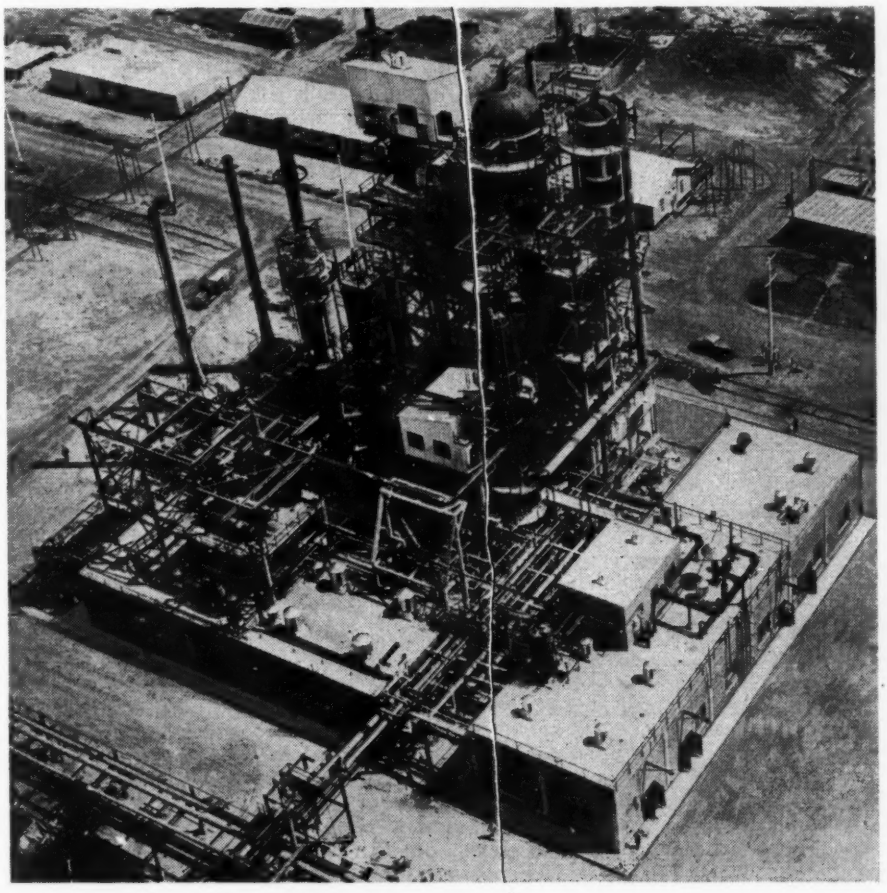
All pumps, compressors and blowers in the unit are steam-driven for reliability and ease of control. The unit itself generates the majority of the steam required for this purpose by recovery of heat from the flue gases and from the hot slurry which has been circulated to de-superheat the reactor vapor.

A new "balanced pressure" design is embodied in the unit. As a result, reactor and regenerator are supported at the same levels; an overall reduction in height has been effected; and standpipe heights have been reduced. The reduced standpipe heights in turn enable a lower pressure drop to be maintained across the slide valves and so eliminate much of the erosion formerly found in these critical catalyst controlling mechanisms. Piping, too, has been simplified, with short direct runs to all major vessels and with only one expansion joint in the whole unit.

The plot plan, arranged in the shape of an elongated oblong, brings the reactor, regenerator, and precipitator into line in a main structure at the extremity of the battery limits. At the opposite face of the oblong, a second row of vessels brings the fractionator and gas recovery equipment into line. This oblong arrangement, open on all four sides with all big vessels on the outside, enabled each vessel to be lifted into position as soon as it arrived on the job site without interfering with other erection proceeding simultaneously.

Design and construction of The Texas Co.'s new Fluid unit at Casper was by M. W. Kellogg Co.





Above—Outside spent catalyst stripper for Fluid "cat-cracker" at Casper, Wyo., refinery of The Texas Co.

Left—New 4000 b/d rated capacity Fluid unit at Casper, Wyo., refinery of The Texas Co.

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SPECIAL APPLICATIONS of ABSORPTION SPECTROSCOPY

By F. W. CRAWFORD
and C. K. BUELL

Research Department
Phillips Petroleum Co.

The purpose of this paper is to describe a number of specialized uses of absorption spectroscopy in the petroleum industry. The methods and procedures presented are not necessarily original, but all have been used to advantage by the authors and their associates.

Infrared absorption spectroscopy has been especially useful for conducting surveys of the composition of the vapor throughout distillation columns. For example, a most satisfactory survey of a 100-plate column may be completed within 24 to 48 hours by two or three persons. Due to the economy of this method, columns operating under a variety of conditions may be studied in considerable detail. Often, as a result of the information gained, a method of column control can be worked out which involves a very simple short-cut analytical method. Several short-cut methods are explained.

A practical analysis for total aromatic content of a stream sample which required only one extinction measurement was worked out. The total aromatic content for each of several samples was determined by the TIWC Referee Method No. 6AR-42, and these values were plotted against the extinctions of the respective samples at 6.15 microns. A satisfactory working curve is given.

Determination of small amounts of impurities in very pure compounds has been accomplished sometimes by careful comparisons of samples from different sources. This involved a qualitative analysis made through a detailed comparison of the spectra, and a very careful quantitative comparison of extinctions of the samples at selected absorption regions.

Employing the foregoing technique, it was also possible to make very precise determinations of the compositions of blends of pure compounds in preparing reference fuels.

EVERY laboratory that has used absorption spectroscopy as an analytical tool has developed specialized applications and short-cut procedures in which the primary objective was to obtain specific information with sufficient precision in a minimum time. This paper was written in order to present and evaluate a few of these developments. Although many of the procedures to be discussed have been used or suggested by others, no attempt has been made to acknowledge the work of individuals or groups because it would be impossible to recognize all of the contributions, formal and informal, that have been incorporated in this paper. However, every analytical procedure discussed here has survived the test of usefulness by the authors and their coworkers.

Distillation Column Surveys

Complete knowledge of the compositions of the vapor and liquid phases at all points in a distillation column is very valuable information but it is also very hard to obtain. The principal difficulty is that so much analytical work is required. In practice, the engineers find that a complete analysis of the vapor every fifth or tenth tray will enable them to calculate any other information needed and this combination of analysis and computation seems to be a very satisfactory compromise in a large number of cases.

Spectroscopic methods of analysis have proved especially suited to the foregoing problem for a number of reasons. The complete set of samples may be taken in small containers practically simultaneously by two or three men and, if desired, the column survey curves can be completed within a day or two. For the information gained, the expenditure of man power is so low that it is practicable to make these detailed studies of column operation under a variety of operating conditions.

A more detailed idea of the nature of the spectroscopic data available may perhaps be gained by a study of Fig. 1, which shows some of the infrared absorption spectrograms of samples obtained in the survey of a column producing pure butadiene. At least one absorption band for each component has been marked in the figure and, by observing the changes in the bands from one spectrogram to the next, it is possible to follow the changes in the concentrations of the various components.

An example of a complete survey of the vapor in a column operating under total reflux is shown in Fig. 2.

In the foregoing examples each sample was subjected to a complete infrared analysis by the straightforward method for multi-component mixtures. A considerable economy in time was effected by analyzing the samples in their order throughout the tower, in that the changes in composition of the samples were small.

Short-Cut Analytical Methods

There are circumstances in which it is possible to analyze for a single component only in a multicomponent mixture and often the operation of a column or other unit can be correlated with the concentration of this key component.

One example of such a situation is furnished in the use of ultraviolet absorption analysis for butadiene in the presence of other C_4 hydrocarbons. In particular, the data for the survey curve of Fig. 3 showing the concentration of butadiene vapor throughout a furfural extractive distillation column were obtained within two or three hours, and thereafter periodic single determinations of butadiene at one selected tray were sufficient to keep the operator informed on the performance of the column.

Inasmuch as butadiene has strong absorption in the ultraviolet over wavelength regions where paraffins and mono olefins do not absorb, the situation is particularly favorable to the application of automatic analyzer-controller devices.

Another example of a useful short-cut method of analysis for a key component can be demonstrated from a stream of C_4 hydrocarbons. Compositions from time to time were determined by multicomponent infrared analysis to be that shown in the first part of Table 1.

At the n-butane absorption band,

TABLE 1—Infrared Analysis of C_4 Stream Short-Cut Method for N-Butane

Sample No.	1	2	3	4	5	6	7	Remarks
Cis-2-Butene	0%	0%	0.6%	0.4%	0%	0%	0%	
1,3-Butadiene	16.6	16.1	15.1	23.2	22.0	11.0	14.4	
1-Butene	50.0	51.2	55.2	54.2	50.3	59.1	47.2	
trans-2-Butene	5.3	5.3	4.4	2.6	2.5	6.8	4.4	
Iso-butylene	9.9	10.9	9.0	10.4	8.2	3.9	3.9	
Iso-butane	5.5	5.2	4.2	5.8	5.5	3.8	4.5	
n-Butane	5.4	5.4	3.7	4.6	9.2	9.2	26.2	Key component
Total Extinction at n-Butane Band	0.084	0.084	0.080	0.088	0.104	0.104	0.166	Ex
Extinction for all but n-Butane	0.060	0.060	0.064	0.068	0.064	0.064	0.052	Ec Average = 0.062
Approximate value n-Butane	5	5	4	6	10	10	24	(Ex-Ec)K
Precise value n-Butane	5.4	5.4	3.7	4.6	9.2	9.2	26.2	from above

TABLE 2—Composition of Samples From Trays X and Y

	Tray X	Tray Y	Equivalent Extinctions at n-Pentane Band
n-Pentane	1.2%	0.0	
2-Methyl-1-Butene	32.4	12.0	13%=1% n-Pentane
1-Pentene	35.1	22.0	4%=1% n-Pentane
Iso-pentane	29.7	64.5	33%=1% n-Pentane
3-Methyl-1-Butene	2.2	0.7	12%=1% n-Pentane

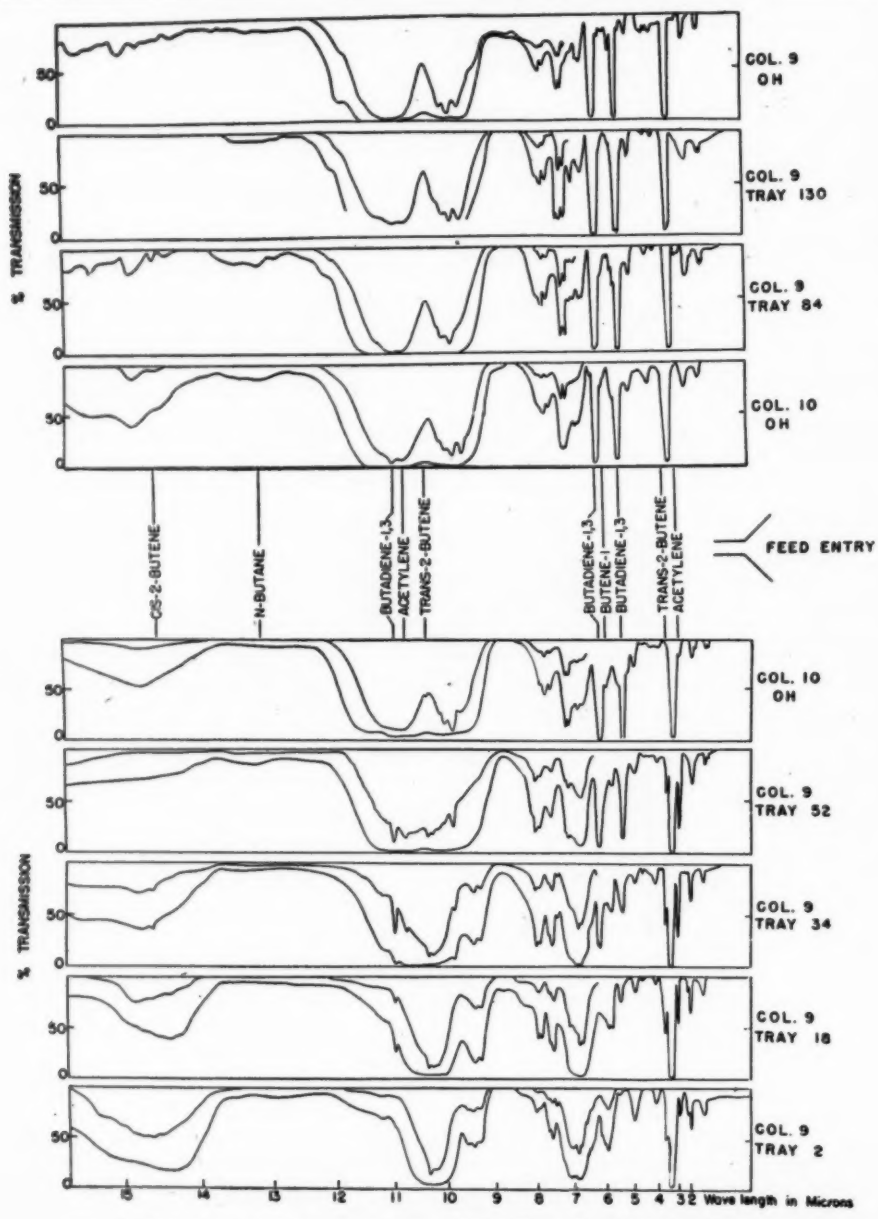


Fig. 1—Infrared absorption spectra of butadiene fractionator tray samples

the measured extinction Ex for all components of the stream has been shown for each sample. Also, the computed value of extinction Ec contributed to Ex by all components except n-butane has been shown for each sample and it is seen that Ec is practically constant. Assuming the average Ec to apply to any sample the approximate value of concentration for n-butane was computed from the simple formula.

Per cent n-butane = (Ex-Ec) K where k is a calibration constant.

Both the approximate and precise values of n-butane contents of the seven samples have been posted in the last two rows of Table 1, and it is obvious that the approximate values are quite satisfactory as a guide to control operations.

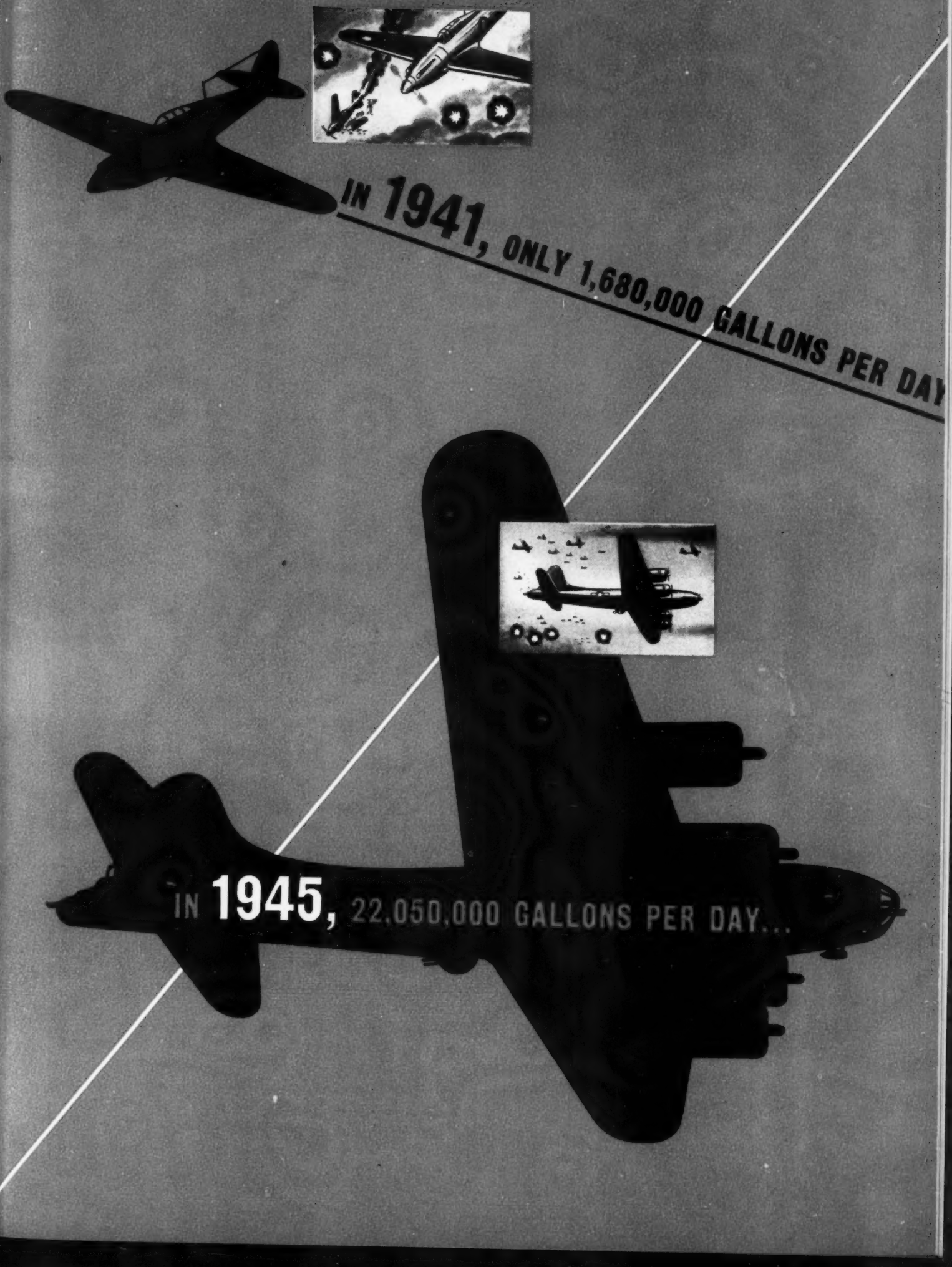
The time required to make a complete analysis of any one sample as shown in Table 1 was one to two hours but the approximate value for n-butane was obtained in about ten minutes. This situation is another in which the application of automatic control should be straightforward.

When the amount of the key component is about one per cent and when the available absorption bands of the key component are not very strong, there is a variation of the foregoing way to analyze a multicomponent mixture for the key component. The procedure required is somewhat like that just described but certain refinements must be made. In essence the procedure is to compare the test mixture with a synthetic mixture whose composition is similar. For example, consider the following circumstances.

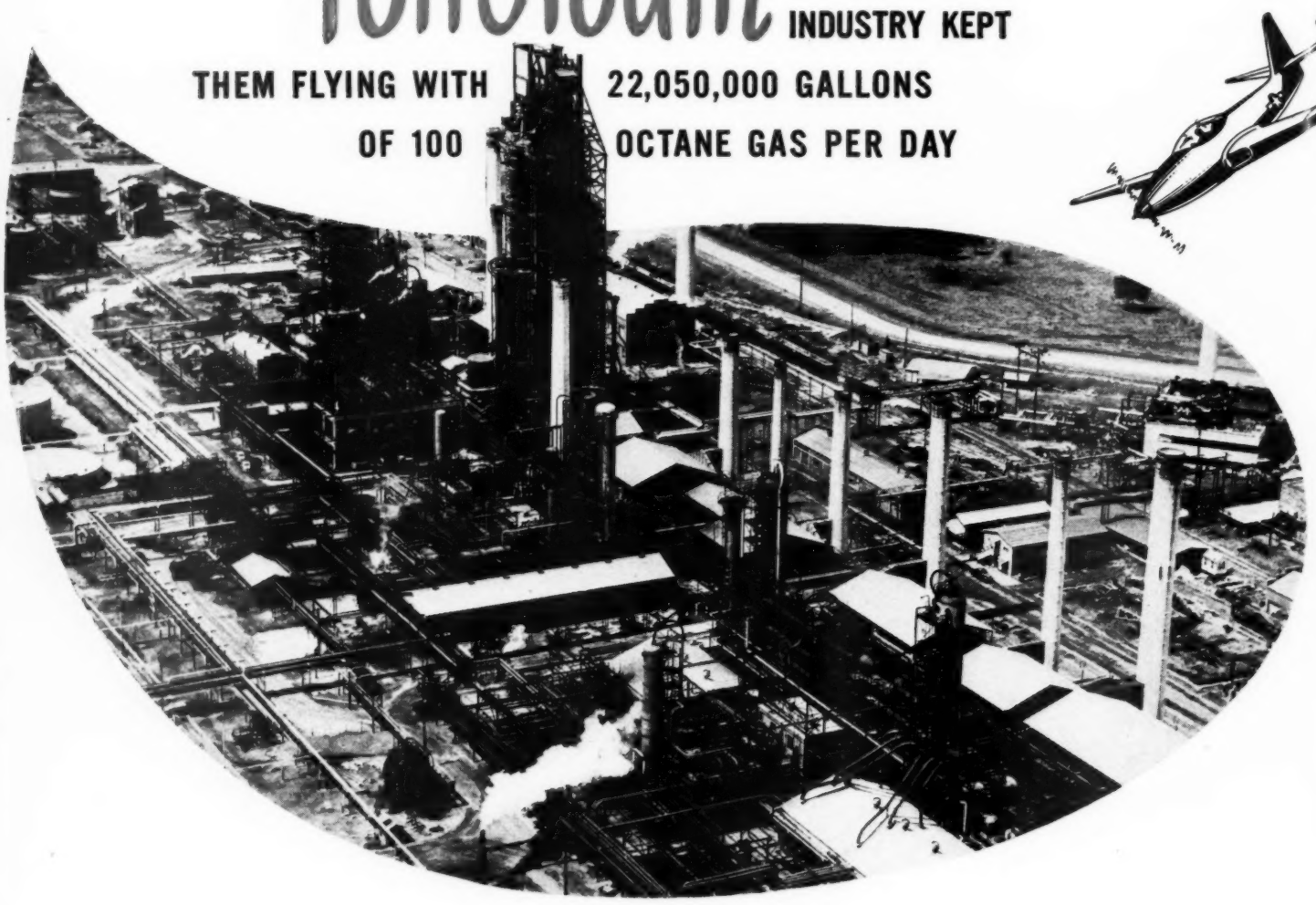
From the data obtained in a column survey it was decided to control a distillation column on the content of n-pentane on Tray X where a typical composition was that shown in Table 2.

To circumvent the problem of synthesizing a reference mixture, a substantial quantity of material was withdrawn from Tray Y in the same column, the analysis of which is presented in Table 2, Col. 2.

Experiment showed that the changes in extinction at the n-pentane band due to changes in the concentrations of the other components would be as listed in Table 2, Col. 3; this informa-


IN 1941, ONLY 1,680,000 GALLONS PER DAY
IN 1945, 22,050,000 GALLONS PER DAY...

Petroleum INDUSTRY KEPT THEM FLYING WITH 22,050,000 GALLONS OF 100 OCTANE GAS PER DAY



Among the many almost unbelievable accomplishments of the petroleum industry, none is greater than the superb manner in which the industry met the wartime demand for aviation gasoline.

In 1941 America's capacity to produce 100 octane gasoline averaged only 1,680,000 gallons per day.

In August 1945—despite the tremendous difficulties of wartime construction . . . the refinery industry reached an output of 22,050,000 gallons of 100 octane gasoline per day.

With the evolution of the jet plane, the petroleum industry also produced the special fuels which made possible speeds of better than 600 miles per hour.

Characteristically, the petroleum industry continued its wartime expansion with further building, ceaseless research and constant product development. In September 1947, the American motorist bought a gallon of gasoline (exclusive of taxes) for little more than half the price he paid in 1920. And today's gasoline averages 75 to 80 A.S.T.M. octane—against 50 in 1920.

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tion being directly available from the spectrograph calibration data. A calibration for n-pentane was then obtained by the addition of five per cent of n-pentane to a portion of the Tray Y sample and the construction of a working curve.

The reference mixture was compared with each sample analyzed, which procedure required about twice the time of the simpler approximation method, but this one had the advantage that it circumvented the errors that often occur due to temperature changes, non-reproducible instrument settings, etc. A constant correction was made for the presence of the other components according to the data in Table 2, Col. 3. Further precision was gained by making a complete analysis of Tray X sample once each day and correcting for any changes in extinction at the n-pentane band due to changes in the other components.

The principal virtues of the foregoing method were that it permitted control on small content of the key component, and it could be set up quickly.

It may be of interest to observe a record of the column performance over a week's time in terms of the n-pentane content on Tray X. This is demonstrated by the graph in Fig. 4.

The question arose as to whether or not it would be possible to develop a quick method of analysis for the total aromatic content of a certain fraction of boiling point range 200 to 320° F. There is one infrared absorption region at approximately 6.15 microns at which the extinction coefficients of several aromatics, except benzene, are of about the same magnitude.

The material in question was a very complex mixture such that complete analysis would have been practically impossible but there were several samples taken at various times during pilot plant runs in which the total aromatic content had been determined by the TIWC-Referee Method No. 6AR-42.

These values of aromatic content were plotted against experimental values of extinctions at 6.15 microns for the available samples and a working curve was drawn as is shown in Fig. 5. It was assumed that the content of aromatic was correct as given and where more than one infrared determination of extinction for a sample was made the points were connected by a line.

The precision of the infrared method would be somewhat dependent on the relative amounts of different aromatics since the various compounds have slightly different extinction coefficients. It would also be strictly dependent on the precision of the Referee Method for initial calibration. Chemists who are familiar with both experimental techniques have estimated that the precisions of the two

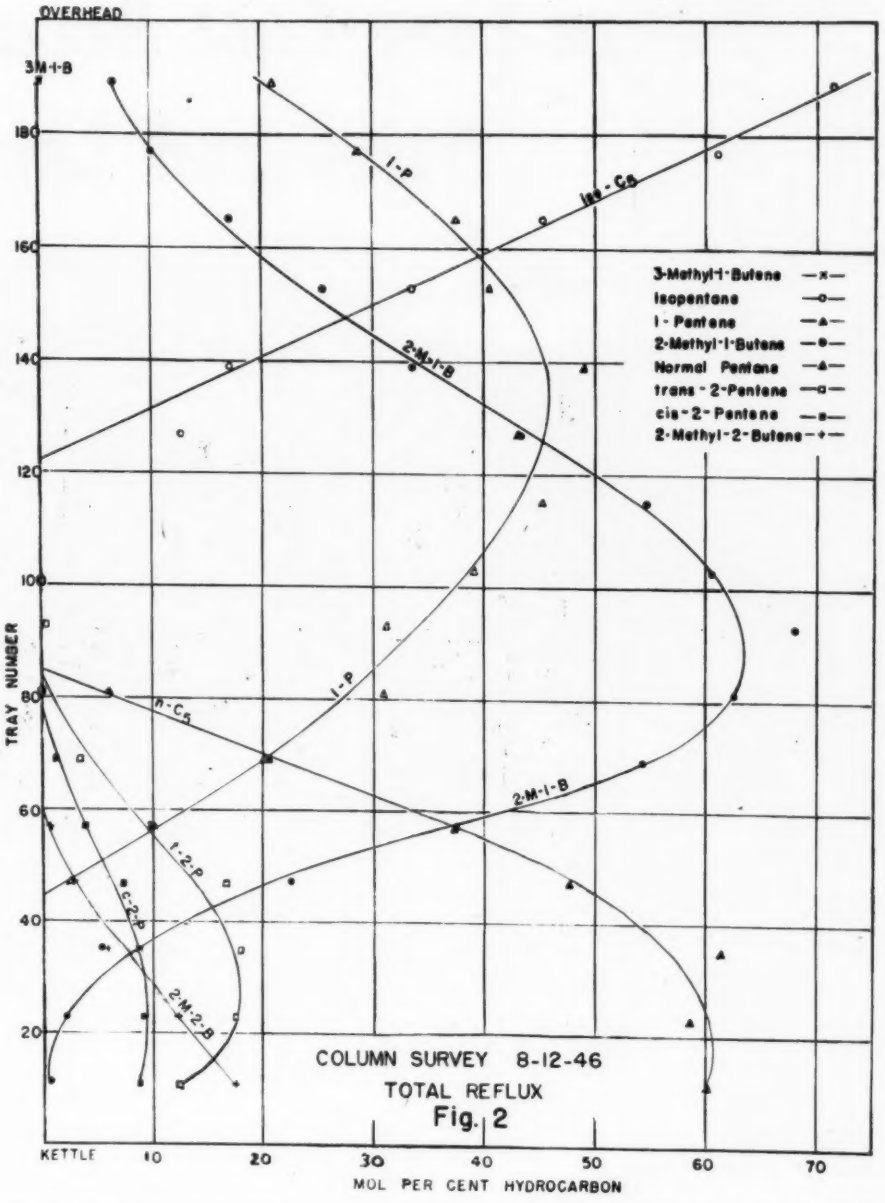

Fig. 2—Example of complete survey of vapor in column operating under total reflux

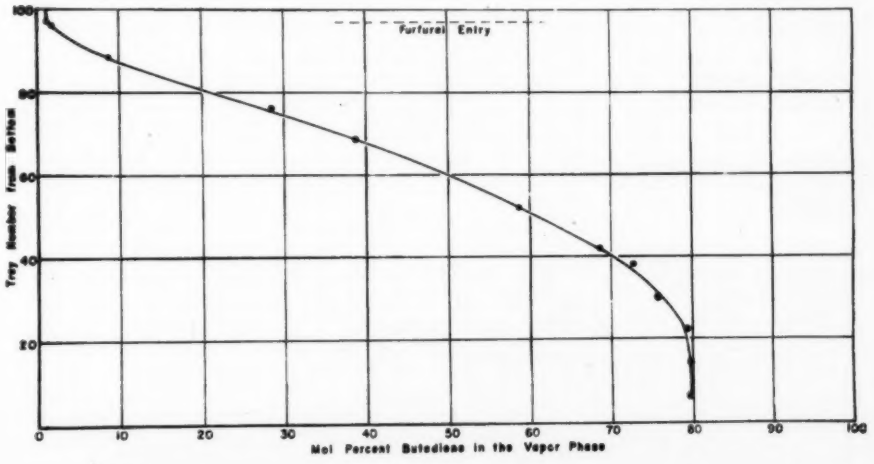

Fig. 3—Butadiene concentration gradient in furfural extractive distillation tower

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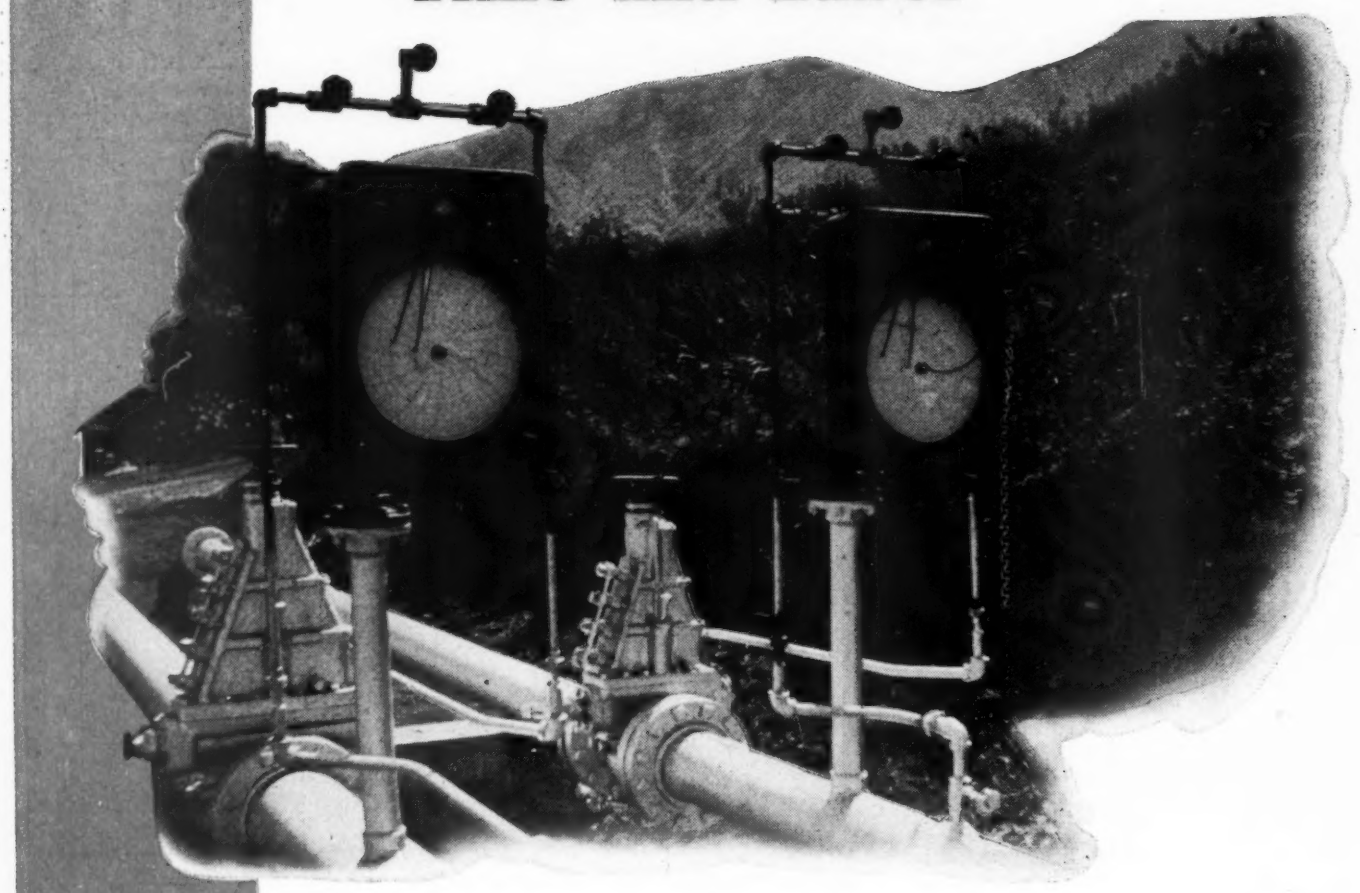

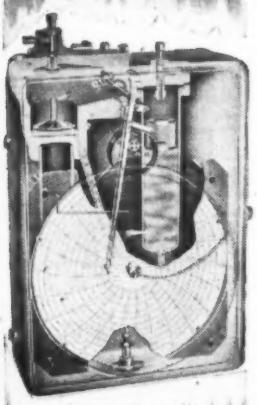

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types of measurement are comparable and hence use of the infrared method is advantageous on the basis of time saved.

Determination of Trace Impurities

A very useful technique for the estimation of trace impurities involves making short scannings of reference samples having known amounts of a certain impurity and estimating the amount of impurity in an unknown from the change in shapes of the curves of the recorded spectra.

A typical problem was the determination of 2-methyl-1-butene in 1-pentane. The very strong Q branch of the 2-methyl-1-butene band at 11.21 microns occurs on the side of the very strong 1-pentene band at 10.94 and thus makes precise extinction measurements difficult. It was found possible to estimate small amounts of 2-methyl-1-butene from the changes in the shapes of the transmittance curves with sufficient precision to referee the production of high purity 1-pentene. Fig. 6 shows comparisons of several samples.

High Precision Methods

The most precise spectroscopic methods are those in which test samples are compared directly with standards. A good example is furnished by some recent experiments conducted to establish the precision that could be expected in day to day analytical work. The samples selected for test were from a large batch of iso-octane and normal heptane which had been blended in the ratio of 60% iso-octane to 40% normal heptane.

Spectrophotometric comparisons were made between the reference fuel blend and three similar blends made in the laboratory from the same base stocks as were used in the large scale blend. In particular, the three accurately compounded mixtures were shown in Table 3. Blending was done with precision gravimetric techniques.

Solution No. 1 was used throughout the experiments as a test sample and was run along with the unknown blend. Some results obtained at the 13.82 micron band of normal heptane and the 8.28 micron band of iso-octane are presented in Table 4. The precision of these measurements as shown in the table can approach ± 0.1% in practice.

TABLE 3—Synthetic Mixtures Used in Analyses

Solution	Iso-pentane Stock	n-Heptane Stock	Accuracy
#1	59.99%	40.01%	±0.03%
#3	59.00	41.00	±0.03
#5	61.00	39.00	±0.03

TABLE 4—Analytical Results

		Composition	Infrared Analysis	No. of Determinations	Average Deviation From Mean
Solution #1	n-Heptane Stock	40.01 ± 0.03%	40.02%	12	± 0.08%
	Iso-octane Stock	59.99 ± 0.03	59.95	5	± 0.04%
		Total	99.97%		
Reference Fuel Blend	n-Heptane Stock		39.98%	6	± 0.06%
	Iso-octane Stock		59.99	3	± 0.18%
		Total	99.97%		

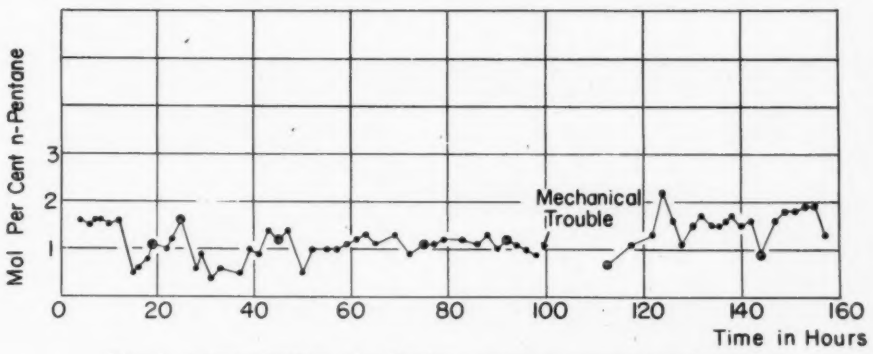

Fig. 4—Concentration of n-Pentane used to control fractionator

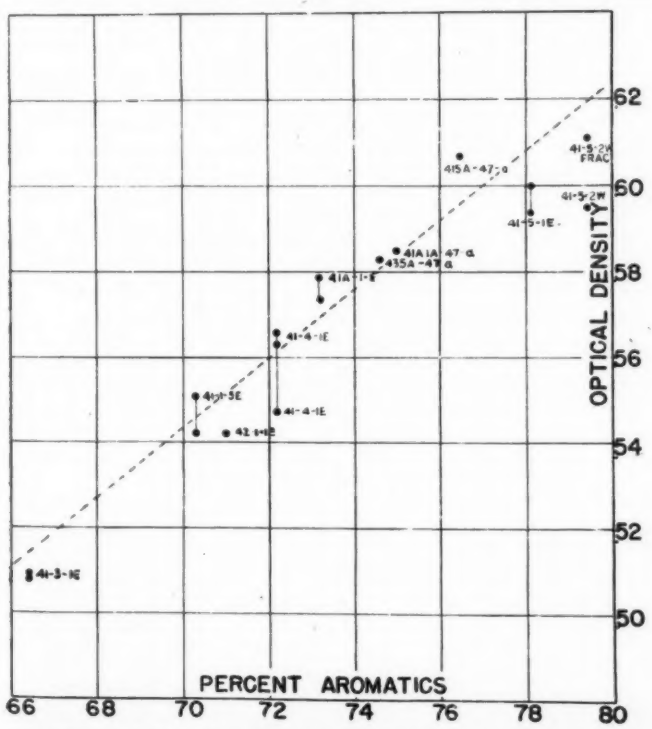

Fig. 5—Infrared working curve for total aromatics at 6.15μ band

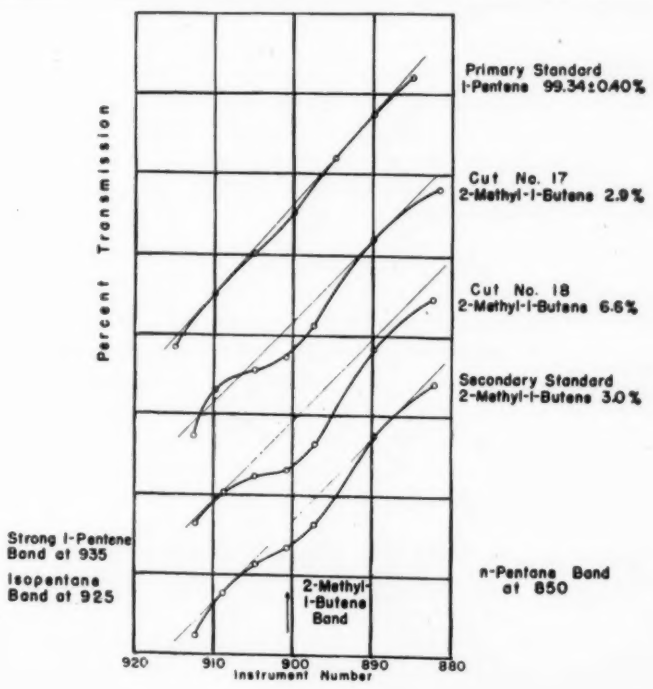

Fig. 6—Check for 2-methyl-1-butene at 11.2μ absorption band

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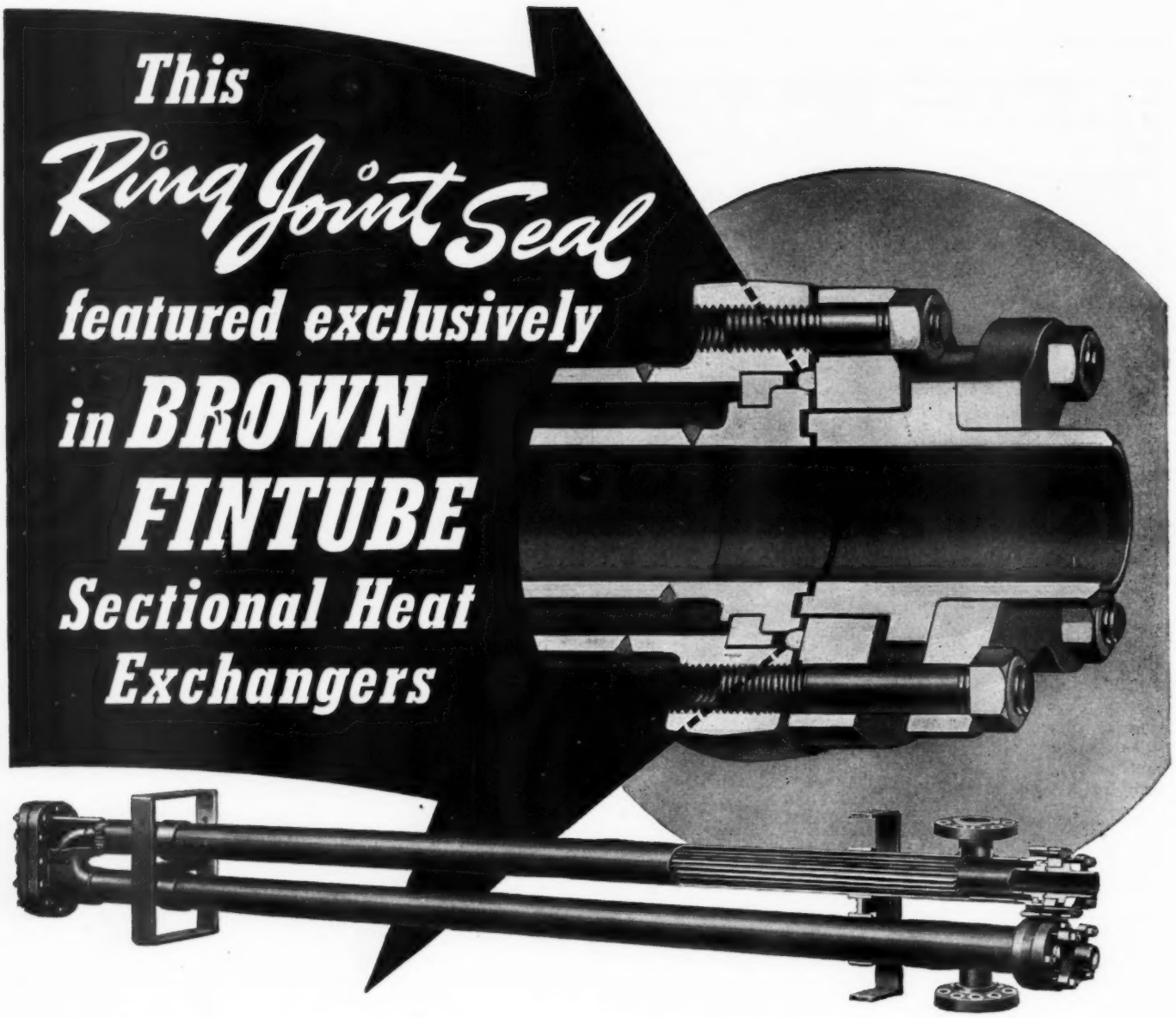

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High-Octane Gasolines by SUSPENSOID Cracking with Synthetic Catalyst

Material in this article is from a paper entitled "The Suspensoid Catalytic Cracking Process", by C. H. Caesar, Imperial Oil Co., Ltd., presented before the Canadian Institute of Chemistry, in Banff, June, 1947.

RECENT studies of the Suspensoid catalytic cracking process carried on at the Sarnia, Ont., refinery of Imperial Oil Ltd., have demonstrated the potentialities of the process for small refineries in making high octane motor fuels. Results of the studies were reported at the meeting of the Canadian Institute of Chemistry in Banff last June.

The development work reported was carried on in a pilot plant especially built for the purpose, which closely reproduced operating conditions in full scale plants. The experimental studies included the use of synthetic cracking catalysts in place of natural clays; charging as feed the total stocks available for motor gasoline production, the resulting distillate representing a refinery pool gasoline; and operations on stocks from various types of crudes to determine the effect of the nature of the crude on the yields and quality of cracked products.

Plant scale Suspensoid cracking has been carried on at the Sarnia refinery as a normal operation for seven years. The Suspensoid process differs from other commercial processes in the use of small quantities of catalyst and in the absence of special reactors. In the operation of the process* powdered catalyst in the form of an oil slurry is charged with the feed to a conventional type of thermal cracking coil. The catalyst is recovered by filtration from the heavy fuel oil bottoms from the fractionating tower, after which it may be regenerated and reused. Between two and 10 pounds of catalyst per barrel of total feed are customarily used.

The report on the pilot plant work on the Suspensoid process, as presented in the paper by C. H. Caesar, of Imperial Oil, Ltd., before the Canadian Institute, is as follows:

Tests with Synthetic Catalysts

"While the product quality obtained in the above refinery operations using natural clays, once through, is adequate for present day requirements, it was realized that future developments would result in a demand for gasoline with octane numbers which could only be obtained by the use of highly active cracking catalyst. Such catalysts, of course, would be regenerated and recycled to the process.

"Experiments in this field were most readily conducted in a pilot plant which was especially built for the purpose, and which closely reproduced the conditions and results obtained in the full scale units.

"Runs in the pilot plant demonstrated that the utilization of fresh 3A catalyst, which is a typical synthetic cracking catalyst commercially available at the present time, would produce gasolines having clear octane numbers approximately two points higher than those obtained when cracking to the same conversion with the natural and activated clays from lube oil contacting. Product distributions at a given conversion were essentially the same using either catalyst. When including non-selective polymer gasoline (produced from the propylene and excess butylenes) gasoline yields of 61% were obtained from Mid-Continent-Louden feed stocks composed of 25% of heavy naphtha and 75% of mixed gas oils. The ASTM Motor method octane number of this gasoline reached 80, and the CFR Research octane number 91, without the addition of tetraethyl lead.

"Regenerated 3A catalysts obtained from commercial Fluid cracking units gave results approaching those of the fresh material, but gave slightly higher yields of gasoline with somewhat lower octane numbers.

"Alternative cracking catalysts which have been investigated to date also tended to produce slightly higher yields of gasoline which had lower octane numbers than those resulting from the use of 3A catalyst. However, the field of preparing catalysts for petroleum cracking is in an active state of development. The results already attained show that the Suspensoid process is sensitive to and will profit by further improvements in catalyst activity.

"As the octane number requirements for motor gasoline increase, the problem of upgrading the light naphtha produced from most crudes becomes increasingly acute. In the case of Suspensoid catalytic cracking, one solution is to include such light naphtha in the total feed to the operation. The results of cracking such a mix are given in Table 1 in which results at two different conversions are reported. In these operations the feed stock was composed of 15% of light naphtha, 25% of heavy naphtha and 60% of mixed gas oils, all from Mid-Continent-Louden stocks. Since this feed represented the total stock available for motor gasoline production, the resulting distillate represented a refinery pool gasoline. As such, unleaded Motor

TABLE 1—Suspensoid Cracking of Mid-Continent-Louden Feed Stocks

Catalyst:	Fresh 3A 10 lb./Bbl.	Feed: API Gravity		40.7°	
Coil Pressure:	600 psig	Naphtha Content		40 LV %	
Feed Rate:	9-10 V/V/Hr.*	Engler Dist. 50% off at		518°F.	
Approximate Coil Outlet Temperature,** °F.	1060		1105		
Yields Excluding Polymer Gasoline					
Gasoline 10 Lb. RVP, 400°F. B.P., Vol. %	56.5		53		
Heating Oil (415-600° Boiling Range), Vol. %	16	(28° API Gravity)	8	(20° API Gravity)	
Fuel Oil (over 600° Boiling Range), Vol. %	8.5	(7° API Gravity)	9	(0.5° API Gravity)	
Excess Butane,*** Vol. %	5		8		
Dry Gas (Propane and lighter), Wt. %	16		24		
Coke, Wt. %	0.8		1.0		
Gasoline Octane Numbers					
ASTM Motor method unleaded	75.0		78.3		
ASTM Motor method +3 cc. TEL	82.9		84.2		
CFR Research method unleaded	86.3		90.0		
CFR Research method +3 cc. TEL	93.7		96.4		
Gasoline Including Polymer—10 Lb. RVP					
Yield on Fresh Feed, Vol. %	62.5		62.5		
ASTM Motor Octane No. unleaded	76.0		81.0		
CFR Research Octane No. unleaded	87.0		91.0		

* Volumes of liquid feed (at 60° F.) per volume of coil above 800°F. per hour.
** Temperature of lead bath at the outlet section of the coil.
*** Excess over requirements to make a 10 Lb. RVP gasoline.

*"Suspensoid Catalytic Cracking", by Robert L. Purvin, PETROLEUM PROCESSING, May, 1947, pg. 328.

octane numbers around 80 and Research octane numbers of 90 (including polymer gasoline) were notably high.

Effect of Feed Source

"The greater part of the research conducted to date has been concerned with the Suspensoid cracking of feed stocks derived from Mid-Continent-Louden crudes. A few runs have been carried out using feed stocks derived from other crudes. These indicate that, as would be expected, the nature of the crude has an appreciable effect on the yields and quality of the cracked products.

"Table 2 illustrates the results of Suspensoid cracking feed stocks from four different crudes, in order of decreasing paraffinicity, at a constant conversion to dry gas. Although the feed stocks varied widely in distillation characteristics, the octane numbers of the unleaded gasolines produced increased in order of decreasing paraffinicity as measured by the characterization factor. Leaded octane numbers showed a similar increase with the exception of the product from Lloydminster feed, which was higher in sulphur content than the other gasolines.

"Although the naphtha content of the feed obviously plays a large part in determining the yield of gasoline at a given conversion, other characteristics of the feed also have an important effect on all the product yields. Sufficient data is not yet available to allow general correlations to be made, so that the results of Suspensoid cracking on unfamiliar feed can only be estimated in a very qualitative manner."

The report on earlier experience by Imperial Oil with Suspensoid catalytic cracking, as presented before the Canadian Institute of Chemistry, brought out that plant scale operation of this process has been carried on since 1940. The catalysts used were natural and activated clays which had been previously used to contact-treat lubricating oils.

Typical operating conditions were coil outlet temperatures of 1045 to 1055°F and coil outlet pressures of 350 to 450 psig. It was found possible to increase the fresh feed rate to the coils over and above that used in the previous thermal cracking operations and still gain an advantage in the octane number of the distillate produced, of approximately 8 points by the ASTM Motor method and 11 points Research.

Appreciable yields of propylenes and butylenes were also produced in these operations. This feature of the process was utilized when the rubber emergency arose during the war, and a Dominion government synthetic rubber plant was built at Sarnia. To supply the maximum quantity of butylenes for this plant, the severity of cracking in the Suspensoid operations was further increased, with coil outlet temperatures ranging from 1080 to 1090°F and at somewhat reduced feed rates. Under these operating conditions increased octane numbers were realized on the gasoline produced.

TABLE 2—Suspensoid Cracking Feed Stocks from Various Crudes

(Feed Rate 9 V/V/Hr., Coil Pressure 600 psig)
Catalyst—Fresh 3A, 10 LB./Bbl.

Crude Source	Mid-Continent-Louden	Jusepin	Colombian	Lloydminster
Feed Inspections				
Gravity °API	40.7	43.3	38.4	34.5
Naphtha Content (400° Engler F. B.P.) L.V. %	40	49	35	27
Engler 50% Point, °F.	509	439	464	484
Characterization Factor	11.9	11.8	11.6	11.5
Conversion to Dry Gas—Wt. % on Feed	15	15	15	15
Yields—Excluding Polymer Gasoline				
Gasoline, 10 Lb. RVP, 400°F. B.P., L.V. %	56.5	63.0	57.4	56.1
Heating Oil (415-600°F. Boiling Range), L.V. %	19.0	13.6	20.2	18.2
Fuel Oil (over 600°F. Boiling Range), L.V. %	8.5	5.0	5.3	6.6
Excess Butane,* L.V. %	5.0	5.6	4.7	7.5
Coke, Wt. %	0.8	1.0	0.8	1.1
Gasoline Octane Numbers				
ASTM Motor method unleaded	74.6	77.4	80.0	80.0
ASTM Motor method +3 cc. TEL	83.0	84.2	85.7	84.0
CFR Research method unleaded	86.0	88.0	91.6	92.5
CFR Research method +3 cc. TEL	93.2	95.0	96.8	96.2
Gasoline Including Polymer—10 Lb. RVP				
Yield—L.V. % on Feed	62.5	68.9	63.1	62.8
ASTM Motor Octane Number unleaded	76.0	78.3	80.5	80.5
CFR Research Octane Number unleaded	87.0	90.6	91.8	92.7

* Excess over requirements to make a 10 Lb. RVP gasoline.

TABLE 3—Comparison of Thermal and Suspensoid Cracking Results

	Thermal Cracking and Reforming	Suspensoid Cracking* Normal Operations	Suspensoid Cracking* Severe Operations
Yields Excluding Polymer Gasoline			
Gasoline, 10 Lb. RVP, 400°F. End Pt., L.V. %	56.1	52.3	53.0
Heating Oil, L.V. %	12.7	12.7	5.1
Fuel Oil, 10° API Gravity, L.V. %	25.6	20.2	19.0
Excess Butane,** L.V. %	1.3	4.5	6.0
Dry Gas (Propane and lighter), Wt. %	6.7	12.5	20.4
Coke, Wt. %	0.3	0.2	0.2
Gasoline ASTM Motor O.N. unleaded	65.4	73.2	75.0
CFR Research O.N. unleaded	71.0	82.0	85.0
CFR Research O.N. +3 cc. TEL	84.8	91.4	94.0
Yields Including Polymer Gasoline			
Gasoline, 10 Lb. RVP, 400°F. End Pt., L.V. %	58.3	58.6	62.8
Heating Oil, L.V. %	12.7	12.7	5.1
Fuel Oil, 10° A.P.I. Gravity, L.V. %	25.6	20.2	19.0
Excess Butane,** L.V. %	Nil	Nil	Nil
Dry Gas (Propane and lighter), Wt. %	5.9	10.5	16.8
Coke, Wt. %	0.3	0.2	0.2
Gasoline ASTM Motor O.N. unleaded	66.6	75.5	78.1
CFR Research O.N. unleaded	72.6	85.1	89.0
CFR Research O.N. +3 cc. TEL	85.6	93.2	96.2

* Catalyst—Natural & Activated Clays from Lube Contacting, 2 lbs./bbl. of feed.
** Excess over requirements to make 10 lb. RVP gasoline.

Typical yields from these operations are given in Table 3, which compares results from thermal cracking with Suspensoid cracking under both the normal and severe operations.

Table 3 brings out that, when polymer gasoline was included in both processes, the Suspensoid operations gave increased yields of gasoline with octane numbers ranging from 9 to 12 points Motor, and 12 to 15 points Research, above those obtained by thermal cracking of similar feed stocks in the same equipment. By increasing the severity of the naphtha reforming operation, it was brought out, the octane numbers of the pool gasoline produced in the thermal case could have been raised 3 to 4 points with a corresponding loss in yield.

The paper brought out that the experience of Imperial Oil Ltd. in full scale Suspensoid catalytic cracking operations the past 6 or 7 years has established the fact it is a low cost operation, well suited to present day requirements. The various mechanical difficulties which arose in the development stage have been eliminated or reduced to such extent that service factors equal to those attained in thermal cracking operations were realized. The octane numbers of the gasolines produced from mixtures of all available feed stocks, and therefore representing refinery pool gasolines, invite comparison with other processes at present available to the industry, it was stated.

The current studies of the Suspensoid process are being continued by the Technical and Research Department of Imperial Oil Ltd., under the direction of Dr. R. K. Stratford, Chief Research Chemist.

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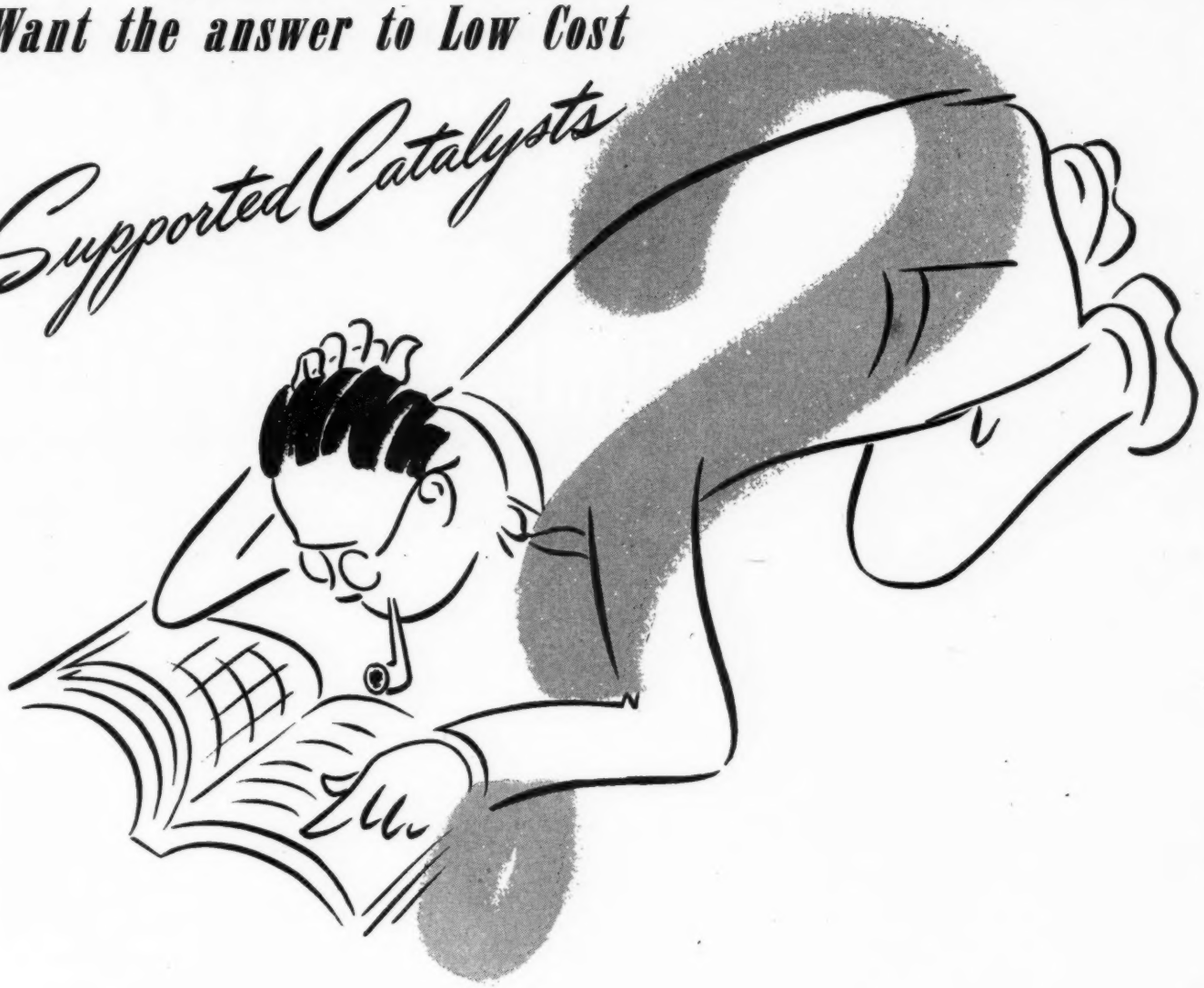

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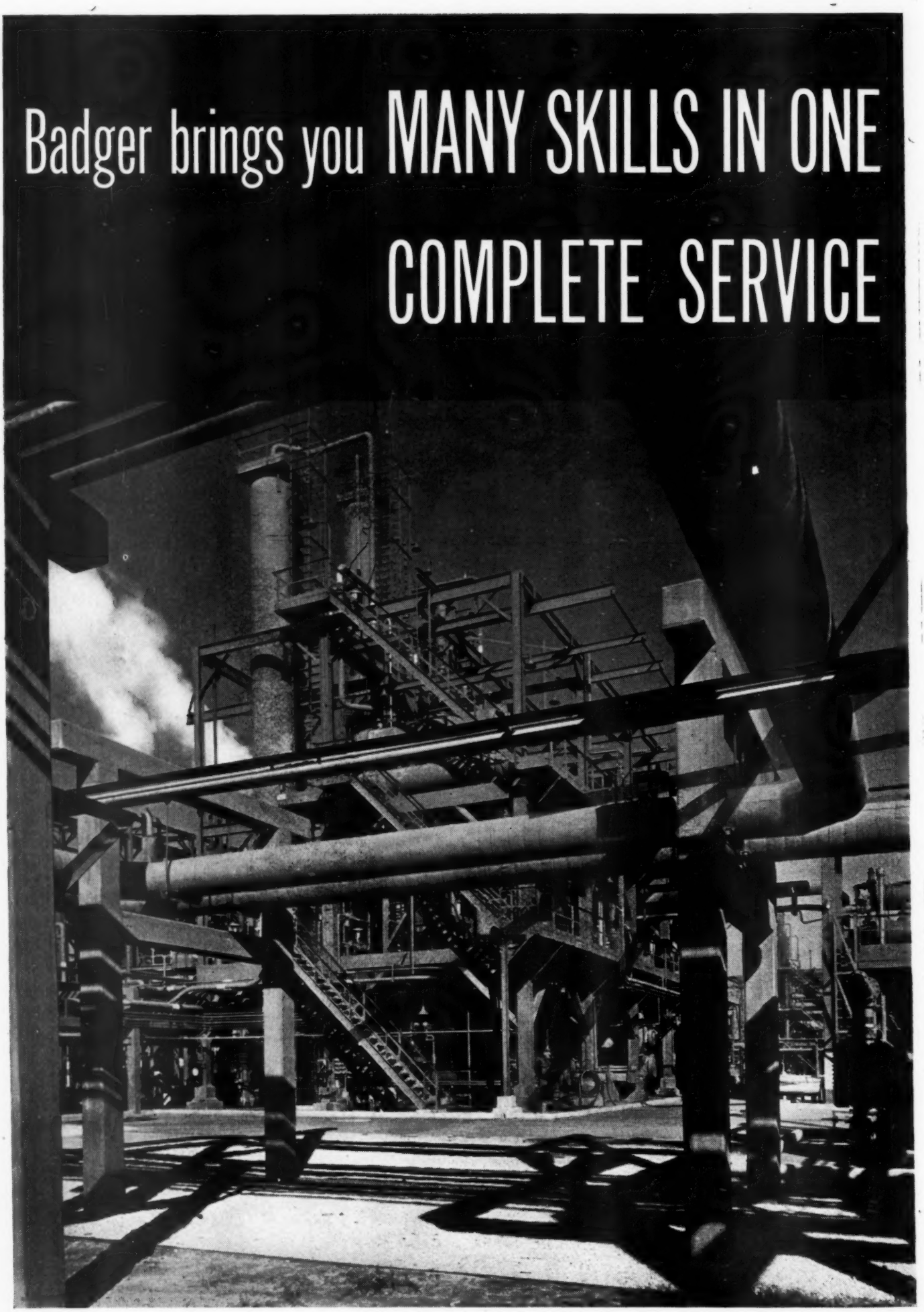

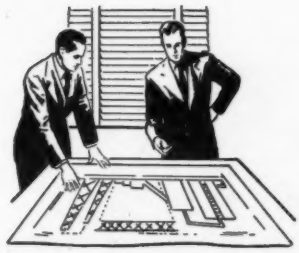

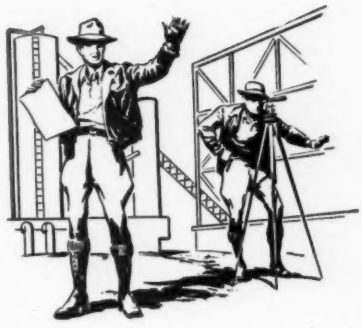

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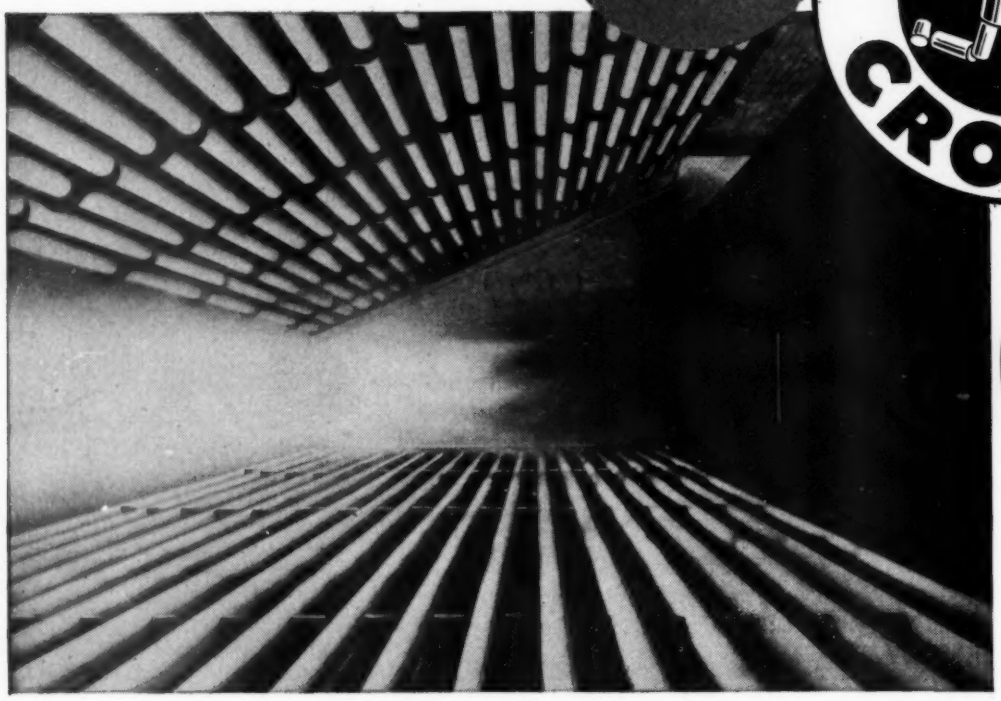

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PAPER

WHERE DOES THE SULFUR GO? *

By M. J. FOWLE and R. D. BENT Atlantic Refining Co., Philadelphia

Data is presented on the distribution of sulfur for various types of processing operations on mixtures of West Texas crudes, ranging from thermally cracking the whole crude to complex distillation, including catalytic cracking. Treating methods studied included mercaptan removal by the Unisol process, sulfuric acid extraction and catalytic desulfurization of napthas and furnace oil. Vapor phase clay treating was found effective in removing the corrosive sulfur content of high-sulfur, straight-run furnace oils.

A summary of product quality by three different methods of crude processing is given. Gasoline produced from West Texas crudes varies from 0.15 to 0.25% sulfur content depending largely on the relative yields of gasoline, furnace oil and residual fuels.

For the individual refinery, the ratio of sweet to sour crude and the ratio of motor fuel to distillate and residual fuels produced will determine the complexity of processing required to meet competitive octane number, sulfur and other performance specifications, it is concluded.

THE current trend in availability of crudes is forcing an increasing number of refiners to process high-sulfur crude oils. When considering the processing of high-sulfur crude, one of the first problems faced is: "Where does the sulfur go?" The answer to this question is essential in order to define and solve process and final product-quality problems.

The aim of this paper is to present information on the distribution of sulfur for various types of petroleum processing. The types of processing considered range from the simplest thermal cracking operation to a more complex combination of distillation; desulfurization; thermal viscosity breaking, gas-oil cracking, and reforming; and catalytic cracking.

The major portion of the information to be presented is based on pilot-plant and plant-scale processing of a collection of crude oils known in the terminology of The Atlantic Refining Co. as West Texas-New Mexico crude. This mixture contans crudes from the Hobbs, Eunice, Wasson, Russell, Slaughter, Seminole, Ector, and Ward-Pecos Pools, with Wasson and Seminole predominating. Boiling-range, specific-gravity, and sulfur-content data for a typical West Texas crude mixture are presented in Fig. 1.

It will be noted that, with the exception of the high-sulfur content and the higher residuum content, this crude is quite similar to East Texas. The West Texas mixture and, more specifically, certain pools in the mix are of interest for the production of lubricants, waxes, and other products. The discussion in this paper, however, is limited to processes for the manufacture of gasoline, and distillate and residual fuel oils.

PRIMARY PROCESSING
Simple Thermal Cracking

The simplest type of processing for the manufacture of gasoline and residual fuel oil is the thermal cracking of the whole crude. The crude oil is charged to the evaporator of a thermal cracking still, and the gas-oil content, together with recycle gas oil, is condensed in the bottom of the fractionator and charged to the cracking furnace. Data for this operation in a deFlorez-type thermal cracking unit are found in Fig. 2.

Here the hydrogen-sulfide production, and the sulfur contents of the gasoline and fuel oil, are related to the yield of fuel oil produced. The crude oil charged in this case averaged 1.77% sulfur. Thus it can be seen that the major portion of the sulfur remains in the fuel oil; and, second, that as lower yield (i.e., of lower gravity) fuels are produced, the gas and distillate as well as the fuel oil increase in sulfur content.

Crude Distillation

Crude distillation units vary widely, particularly with respect to the pressure of operation and the number of stages of vaporization employed. One factor to be considered when planning the distillation of high-sulfur crude is the effect of operating temperature on the decomposition of sulfur compounds. This point is illustrated in Fig. 3, which is taken from plant and pilot-plant data on the distillation of

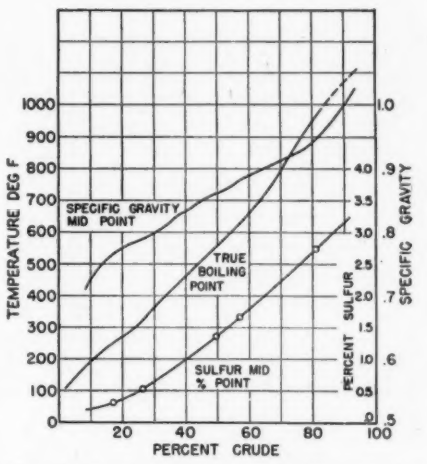


Fig. 1—Boiling range, specific gravity and sulfur distribution for a typical West Texas crude oil

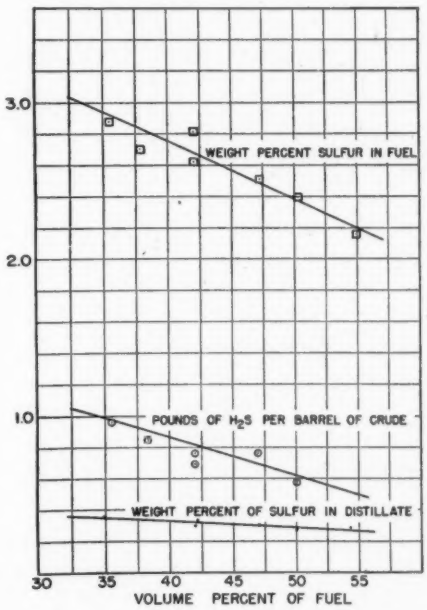


Fig. 2—Product sulfur contents for thermal cracking of West Texas crude oil

* Presented before Division of Refining, 27th Annual Meeting of the American Petroleum Institute, Chicago, Nov. 11, 1947.

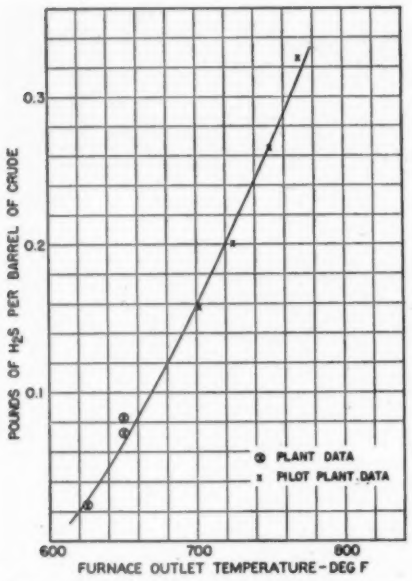


Fig. 3—Relation between furnace conditions and decomposition of sulfur compounds for Texas oil distillation

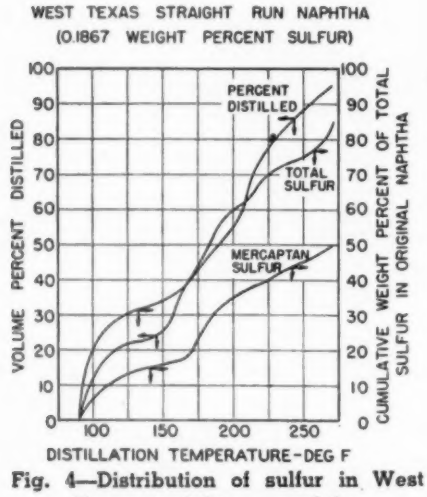


Fig. 4—Distribution of sulfur in West Texas straight-run naphtha

West Texas crude. This curve emphasizes the increased decomposition to be expected when higher pipe-still temperatures are employed. The hydrogen-sulfide production is important from the standpoint of equipment corrosion. It should also be borne in mind that the mercaptan contents of the distillate products increase with increasing temperature.

The distribution of mercaptan sulfur and total sulfur for a typical West Texas light straight-run naphtha, produced at a 680° F. pipe-still temperature, is presented in Fig. 4. Mercaptan sulfur accounts for more than half of the total sulfur in this stock.

Viscosity Breaking

Viscosity-breaking operations are closely associated with primary distillation, and mild viscosity breaking is extensively used to produce gas-oil feed for catalytic and thermal cracking operations. Sulfur-distribution data for a mild viscosity-breaking-vacuum-flashing operation will be found in Fig. 5. The most outstanding point in the viscosity breaking of high-sulfur stocks is the high-sulfur content of the naphtha produced. Fortunately the yield of this gasoline can be held down to 5 to 10% by employing mild cracking conditions. Fuel-oil yield is reduced by employing a vacuum-flash operation. Sulfur-distribution data for a moderately high-sulfur viscosity-breaker gasoline are shown in Fig. 6.

Reforming

Thermal reforming of straight-run naphtha accomplished about a 45% desulfurization, as shown in Fig. 7. The extent of desulfurization varies somewhat with the cracking severity, but there were not sufficient data available to include this factor. The

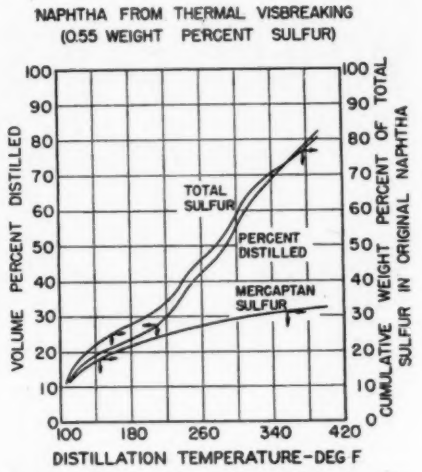


Fig. 6—Distribution of sulphur in viscosity-broken gasoline

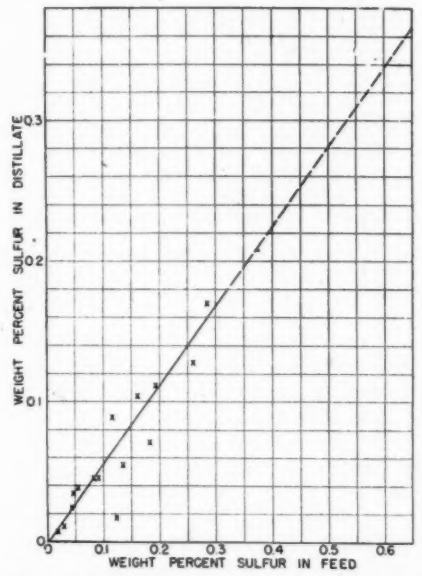


Fig. 7—Relation between feed and product sulfur contents for reforming straight-run naphtha

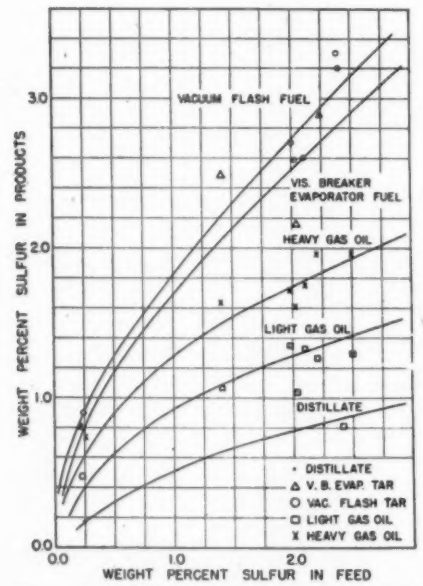


Fig. 5—Sulphur content of products from vis.-breaking of high-sulfur stocks

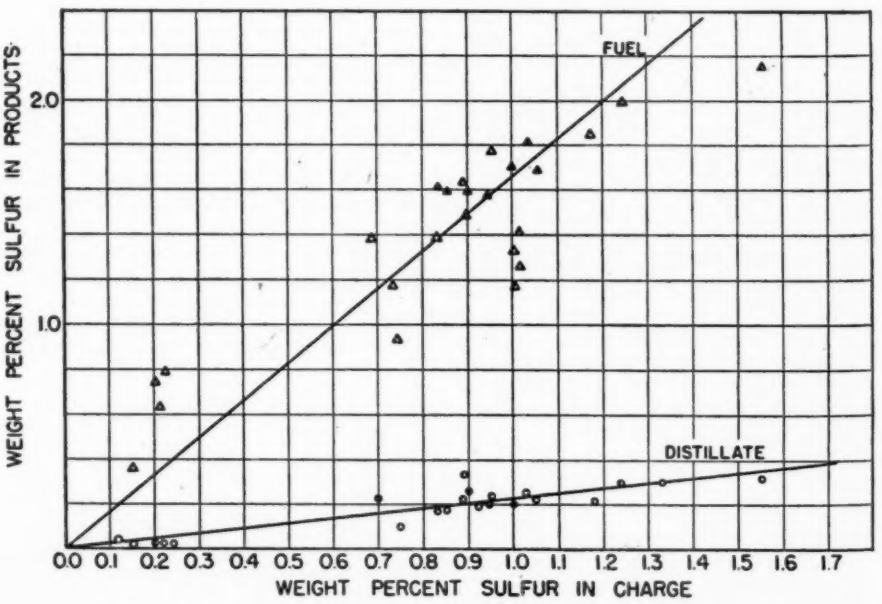
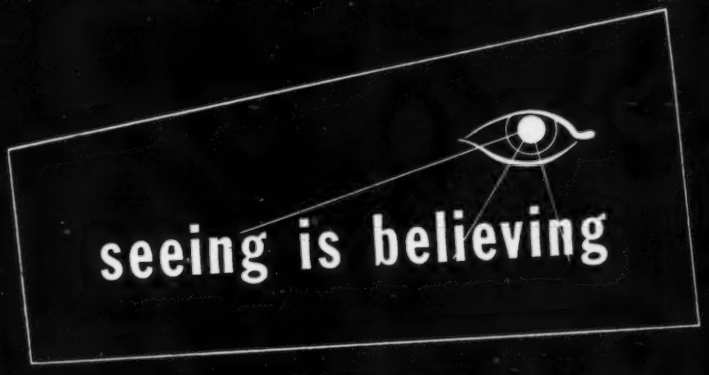

WEIGHT PERCENT SULFUR IN PRODUCTS
FUEL
DISTILLATE
2.0
1.0
0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7
WEIGHT PERCENT SULFUR IN CHARGE

Fig. 8—Sulfur content of products from thermal cracking of gas oils



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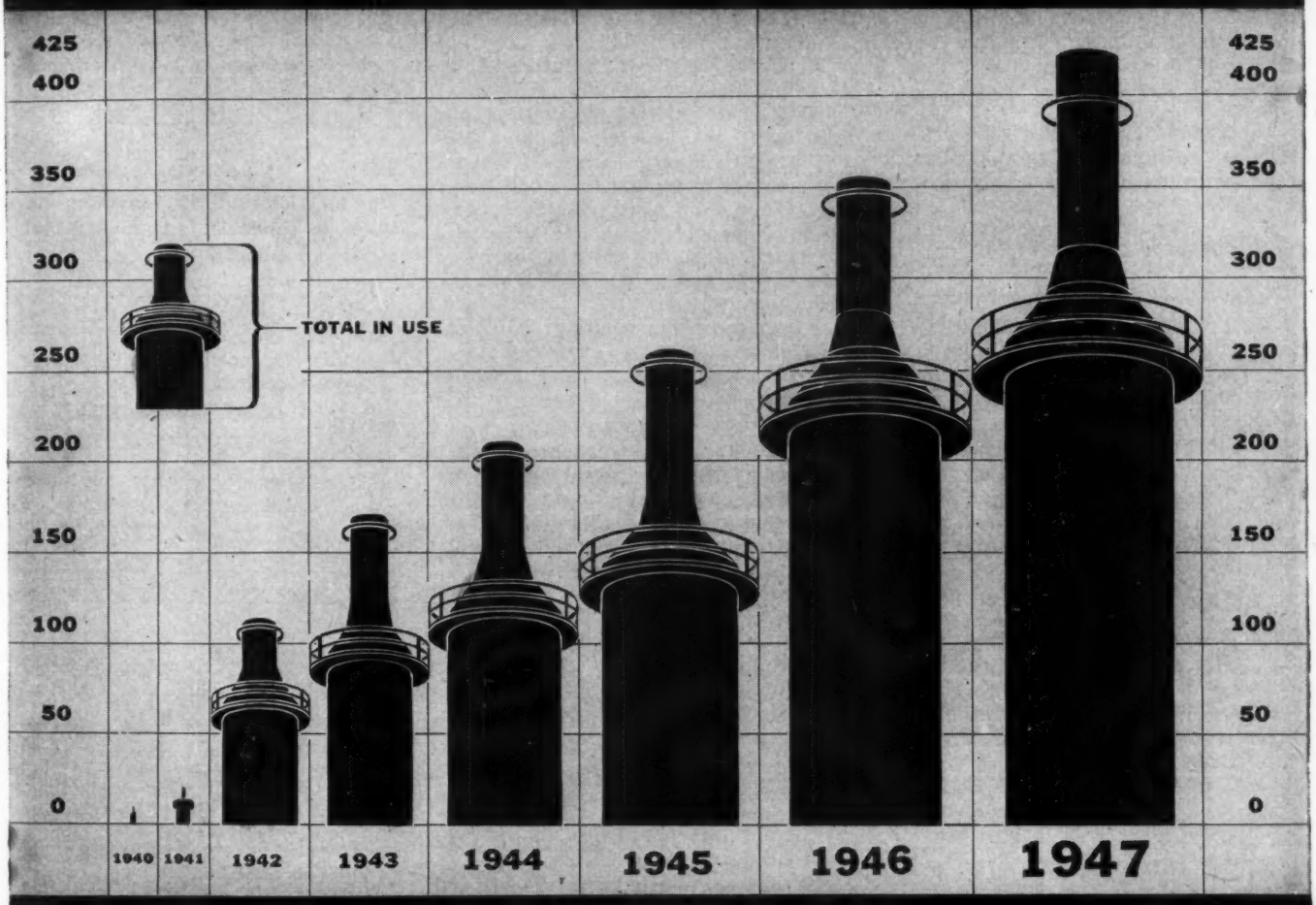
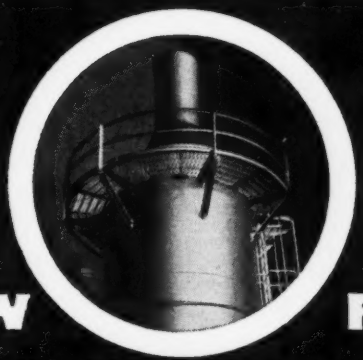


PETR CHEM



ISO-FLOW FURNACES

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data presented represent an average cracking severity equivalent to 10% C_3 and lighter gas production.

Thermal Gas-Oil Cracking

As in the case of crude-oil cracking, Fig. 8 shows that a large portion of the sulfur in recycle gas-oil cracking remains in the fuel oil. The distribution of total sulfur and mercaptan sulfur, with boiling range for a typical thermal cracked naphtha, is illustrated in Fig. 9. Mercaptans represent about 50% of the total sulfur content up to a 300° F. boiling point. The high-boiling fractions contain increasingly greater quantities of sulfur compounds, very little of which is of mercaptan type.

Catalytic Cracking

Sulfur distribution for catalytic cracking differs considerably from that for thermal operations. The three chief differences are: An outlet exists for sulfur to leave the system in the regenerator flue gas; no such outlet exists in thermal cracking. Little or no mercaptan sulfur is produced; catalytic gasolines range from zero to 0.01% by weight of mercaptan sulfur. Polymerization of sulfur compounds is not a factor as in thermal cracking, and cycle-stock fractions do not contain significantly more sulfur than corresponding fractions in the feed; apparently the sulfur which is polymerized tends to leave the system as coke on the catalyst.

Available sulfur-distribution data for fluid catalyst cracking of a wide variety of stocks with synthetic catalyst have been correlated, and are presented in Figs. 10 to 14, inclusive. Per cent conversion, feed-stock boiling range, and operating temperature have been considered as variables. No trend in sulfur distribution was found for a range of 800 to 970° F. operating temperature. There are doubtless a number of other variables such as the activity and contamination of the catalyst, but a greater range of data will be necessary to determine such factors.

Fig. 10 presents an interesting relationship between per cent by weight of coke, feed boiling range, and the proportion of the feed-stock sulfur leaving in the flue gas. For normal boiling-range feed stocks with a 50% point at 650° F. or higher, 10 to 20% of the feed sulfur is discharged in the flue gas. For lower boiling feeds, increasingly greater portions of the feed sulfur follow this route. The sulfur-distribution correlation presented in Fig. 11 was developed for normal boiling-range feed stocks, viz., with the 50% point at 650° F. and higher. It should be noted that the sulfur data are presented in a different manner from those in the preceding charts; i.e., per cent by weight of the total feed sulfur in the individual products is used rather than the sulfur content of the product. Figs. 12 and 13 indicate the prediction accuracy of the sulfur-distribution correlation. The correlation in Fig. 11 is based on cutting a 390° F., 90% point catalytic gasoline. The variation of gasoline sulfur content

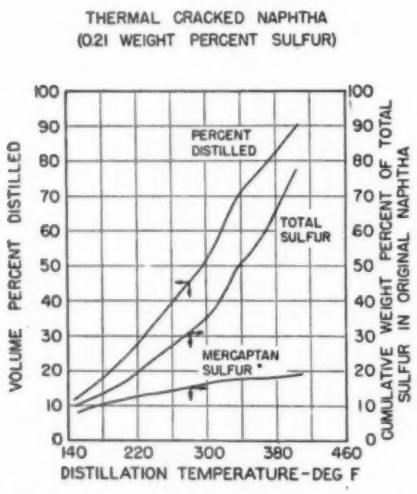

Fig. 9—Sulfur distribution for thermally-cracked gasoline

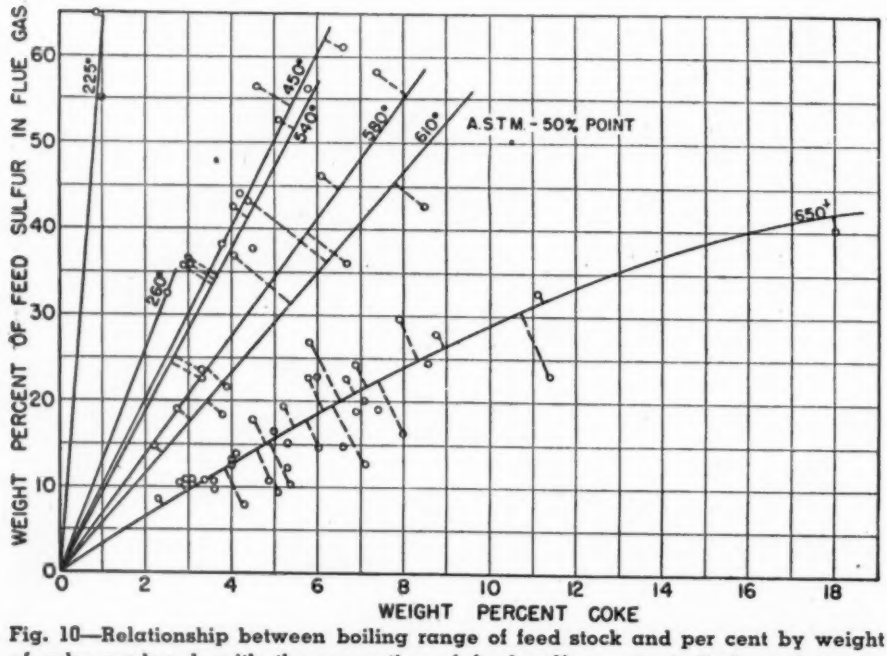

Fig. 10—Relationship between boiling range of feed stock and per cent by weight of coke produced, with the proportion of feed sulfur content discharged in the flue gas of a Fluid catalyst cracking operation

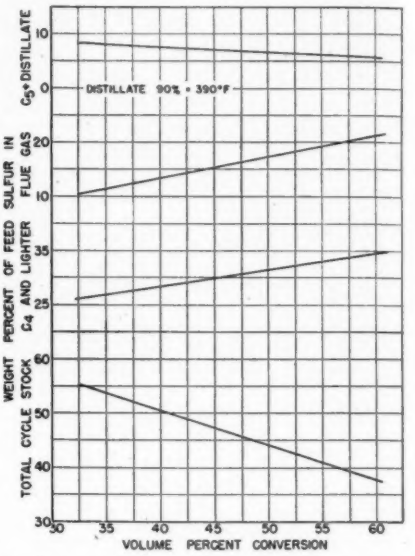
C5+ DISTILLATE
DISTILLATE 90% - 390°F
WEIGHT PERCENT OF FEED SULFUR IN C4 AND LIGHTER FLUE GAS
TOTAL CYCLE STOCK
VOLUME PERCENT CONVERSION

Fig. 11—Correlation for distribution of sulfur in products from Fluid catalyst cracking

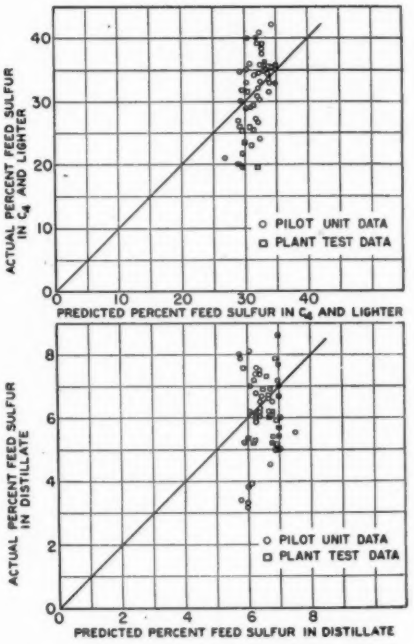
ACTUAL PERCENT FEED SULFUR IN C4 AND LIGHTER
PREDICTED PERCENT FEED SULFUR IN C4 AND LIGHTER
PILOT UNIT DATA
PLANT TEST DATA
ACTUAL PERCENT FEED SULFUR IN DISTILLATE
PREDICTED PERCENT FEED SULFUR IN DISTILLATE

Fig. 12—Correlation of predicted with actual sulfur distribution in C_4 and lighter, and in distillate from Fluid cracking

with changes in cut point is shown in Fig. 14.

Total sulfur distribution for a catalytically cracked gasoline is shown in Fig. 15. No mercaptan-distribution data are included, as the mercaptan content is negligible.

TREATING OPERATIONS

From the sulfur standpoint, treating of gasoline and distillate fuels is carried out for one or more of the following reasons:

1. Reduction of total sulfur content.
2. Removal of corrosive sulfur compounds.
3. Improvement of odor.
4. Improvement of tetraethyl-lead susceptibility.

The oldest treating process still in widespread use is the doctor process for the oxidation of mercaptans to disulfides. This process accomplishes an improvement in odor and corrosive sulfur content with no sulfur reduction, unless practiced in conjunction with vacuum rerunning. Numerous other processes are in general use to accomplish the same purposes as doctor sweetening. Generally, sweetening causes a loss in clear octane number with no change in tetraethyl-lead susceptibility.

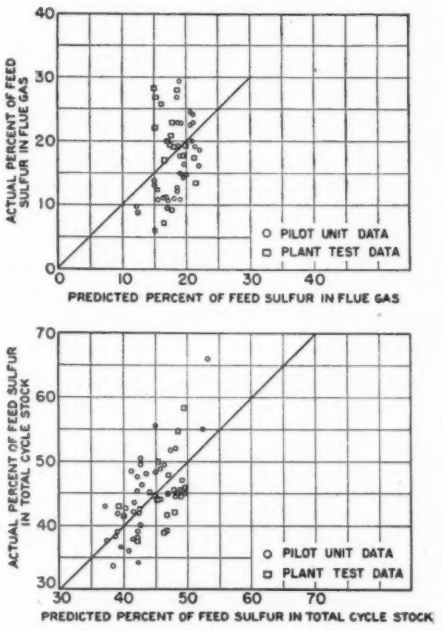
ACTUAL PERCENT OF FEED SULFUR IN FLUE GAS
PREDICTED PERCENT OF FEED SULFUR IN FLUE GAS
O PILOT UNIT DATA
□ PLANT TEST DATA
ACTUAL PERCENT OF FEED SULFUR IN TOTAL CYCLE STOCK
PREDICTED PERCENT OF FEED SULFUR IN TOTAL CYCLE STOCK
O PILOT UNIT DATA
□ PLANT TEST DATA

Fig. 13—Correlation of predicted with actual sulfur distribution in flue gas and in total cycle stock from Fluid cracking

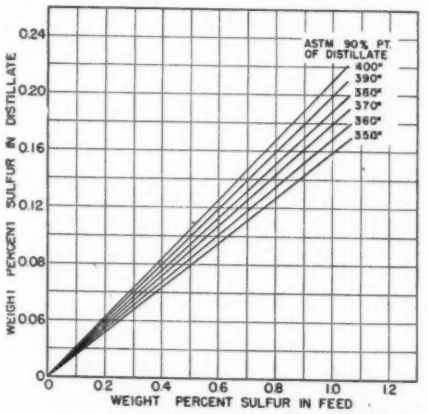

Fig. 14—Effect of cut point on sulfur content of catalytically-cracked gasoline

Mercaptan Extraction

High-sulfur, straight-run and thermally cracked gasolines contain appreciable mercaptan sulfur content; and most modern refineries practice some form of mercaptan extraction, primarily for the improvement in tetraethyl-lead susceptibility and the avoidance of the octane-number loss which takes place when sweetening. The simplest type of mercaptan-extraction process is a counter-current regenerative extraction with aqueous caustic. Such processing accomplishes a 25 to 60% mercaptan removal, depending on the feed stock (proportion of low molecular-weight mercaptans) and the equipment. More advanced mercaptan-extraction processes employing selective solvents, such as the Unisol process(1) and others,(2) accomplish 95% or more extraction of mercaptans. Mercaptan extraction meets all four treating aims listed previously herein. In general, mercaptan extraction of higher boiling distillate fuels is not very effective.

Acid Treating

Sulfuric-acid extraction of sulfur compounds is extensively practiced, particularly for high-sulfur thermally cracked gasoline. Such processing generally necessitates a rerunning operation to remove the polymers formed. It has been found that, by combining sulfuric-acid treating with doctor sweetening and low-temperature vacuum rerun, the sulfur removal can be increased considerably over conventional acid treating. This

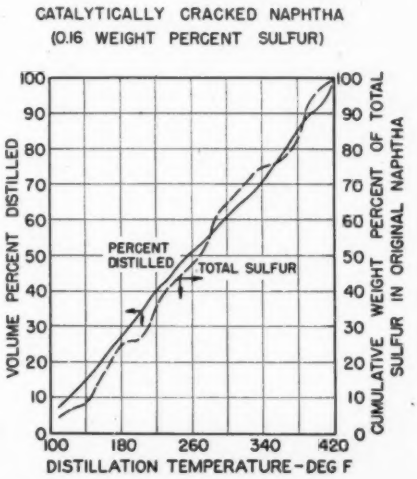
CATALYTICALLY CRACKED NAPHTHA
(0.16 WEIGHT PERCENT SULFUR)
VOLUME PERCENT DISTILLED
CUMULATIVE WEIGHT PERCENT OF TOTAL SULFUR IN ORIGINAL NAPHTHA
PERCENT DISTILLED
TOTAL SULFUR
DISTILLATION TEMPERATURE - DEG F

Fig. 15—Sulfur distribution for catalytically-cracked gasoline

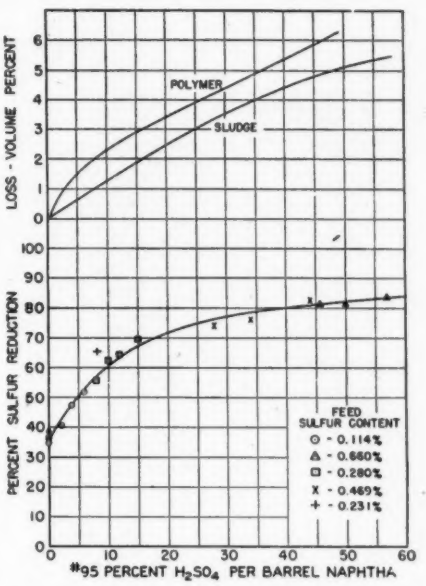

Fig. 16—Yield and sulfur removal on thermally-cracked gasolines for caustic washing, acid treating and doctor sweetening in a low-temperature rerun system

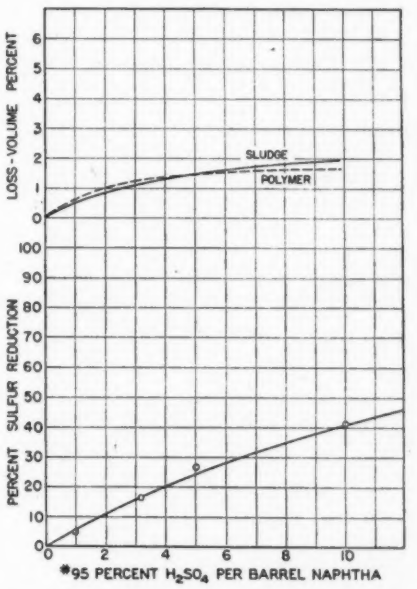

Fig. 17—Yield and sulfur removal on catalytically-cracked gasoline for caustic washing, acid treating and doctor sweetening in a low-temperature rerun system

increase in sulfur removal results from the fact that the disulfides formed are much higher boiling than their corresponding mercaptans.

Fig. 16 presents data on yield loss to sludge, polymer production, and sulfur removal for a treating system consisting of caustic washing, acid treating, doctor sweetening, and steam and vacuum rerunning. It is notable that, for the typical thermally cracked stocks treated, approximately 35% of the sulfur is removed by caustic washing, doctor sweetening, and low-temperature rerun. Thus, if an acid-

Meet the Authors

Mr. Fowle

M. J. Fowle graduated from University of Wisconsin in 1929 with a B. S. in chemical engineering. Prior to going with Atlantic Refining Co. in the research and development department in 1934, he did experimental work for Socony-Vacuum and duPont. At present he is divisional director of research and development work on motor fuels and related products for Atlantic Refining.

Mr. Bent

R. D. Bent graduated from University of Pennsylvania in 1935 with a B. S. in chemical engineering. Employed by Atlantic Refining since graduation, he is now assistant in charge of development work.

treating rerun system is justified for reasons other than sulfur removal (for gum reduction or improvement of stability), it is well worth considering the economics of including a doctor-sweetening and low-temperature-rerun step.

When rerunning doctor-sweetened gasolines, temperatures should be held below 215° F., and hold-up time at 215° F. minimized. It can also be seen in Fig. 16 that sulfur removal increases very slowly above about 70% removal and 20 lb of acid per barrel. For greater removal, higher strength acid and/or low-temperature treating must be considered.(3)

Fig. 17 contains data for a similar treating system on a Fluid catalytic cracked gasoline of 0.24% by weight of sulfur content. Here it is seen that the inclusion of the doctor step is ineffective in removing sulfur, because the gasoline contains very little mercaptan.

Catalytic Desulfurization—Naphthas

The catalytic conversion of sulfur compounds in straight-run gasolines to hydrogen sulfide by passing the vaporized product at elevated temperatures over clay, bauxite, or other catalysts has been practiced for many years—primarily for the improvement of tetraethyl-lead susceptibility. In Fig. 18 the sulfur content of treated naphtha is plotted against clay life for an operation treating 300° F-end-point West Texas straight-run naphtha over Attapulgus 30-to-60-mesh clay catalyst at 750° F. and 1.1 space velocity. The average desulfurization obtained was 83%. The average gain in tetraethyl-lead susceptibility is from 0.9 to 1.9 Hebl slope.(4) The gas loss for the operation is 0.4% by weight, and the gain in clear octane number varies from 0.2 to 0.6 octane number.

Greater gains in clear octane number can be obtained by employing more severe conditions or more active catalysts; but such operations fall in the catalytic reforming category, and involve a much greater yield loss to gas, coke, and polymers. In addition, the catalyst is rapidly deactivated by coke deposition, requiring regeneration.

Catalytic desulfurization has little or no application to thermally cracked stocks. Experience has shown that, with such stocks, the maximum desulfurization obtainable is limited to mercaptan and disulfide sulfur (freshly cracked stocks contain no disulfide). Thus, for thermally cracked stocks, mercaptan-extraction processes achieve the same desulfurization obtainable by catalytic desulfurization without the attendant yield losses to gas, polymers, and coke. These losses are occasioned by reaction of the unsaturated hydrocarbons which, in turn require frequent regeneration of the catalyst.

Catalytic Desulfurization Furnace Oil

High-sulfur furnace oils contain corrosive sulfur compounds which cause considerable difficulty with corrosion and plugging in domestic burner systems. These compounds are difficult to remove by conventional doctor treating, acid treating or caustic washing. It has been found that vapor-phase clay treating, under conditions similar to those employed for naphtha desulfurization is very effective in reducing the corrosive sulfur content of high-sulfur straight-run furnace oils. Data for the desulfurization of an 0.85-percent sulfur-content furnace oil are plotted against clay life in Fig. 19. Based on the hydrogen sulfide produced, the extent of the desulfurization averages about 10% removal.

From the mercaptan-content data, it may be concluded that the sulfur removed is largely mercaptan sulfur. The "four-day iron-copper stability" is the value obtained by the Atlantic chemical stability of furnace-oil test method.(5) Correlation with burner test work, storage tests, and field experience has shown that a value of 15 is acceptable. It should be noted that both the chemical stability and the mercaptan content break at about 4,500-bbl.-per-ton clay life.

Greater desulfurization of furnace-

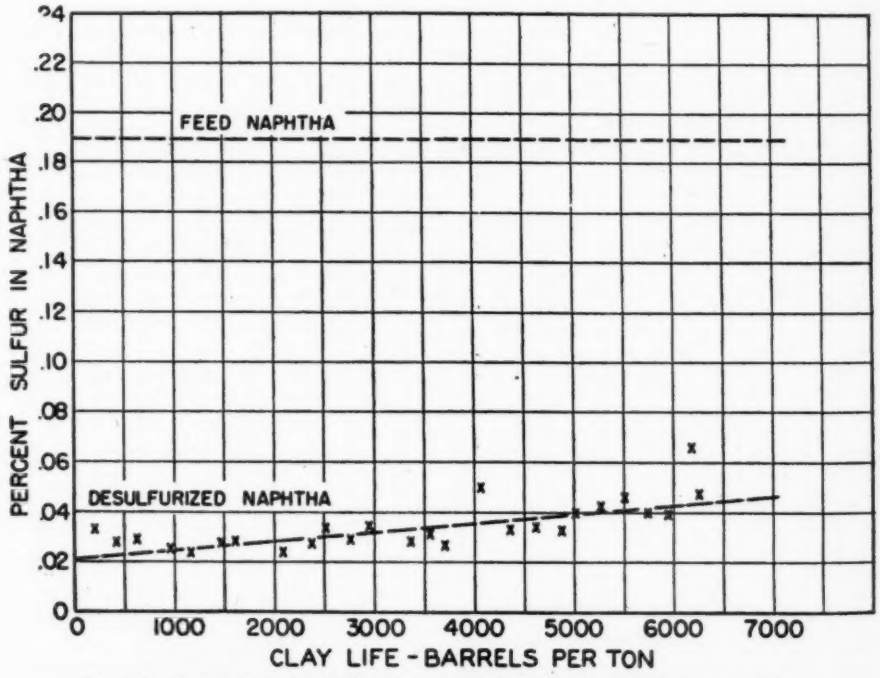

Fig. 18—Catalytic desulfurization of West Texas straight-run naphtha

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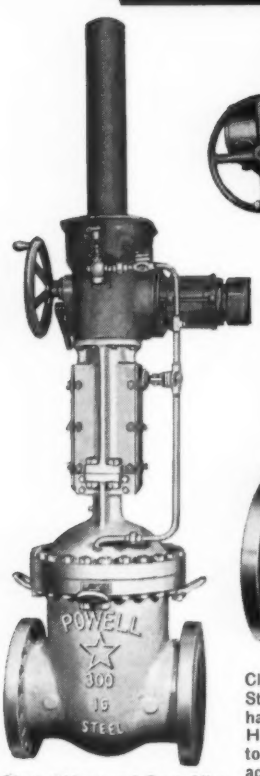
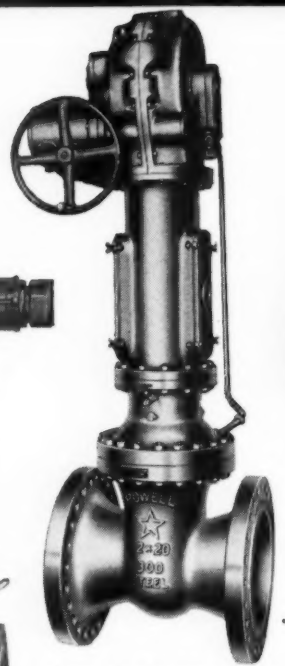


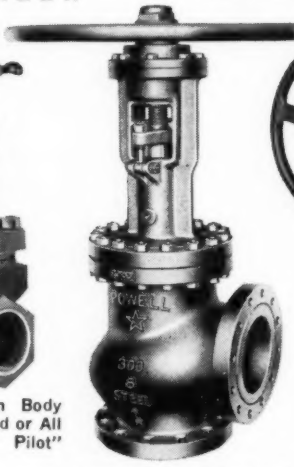
Class 300-pound Cast Alloy Steel Gate Valve with automatic steam sealing mechanism and explosion proof electric motor operator.



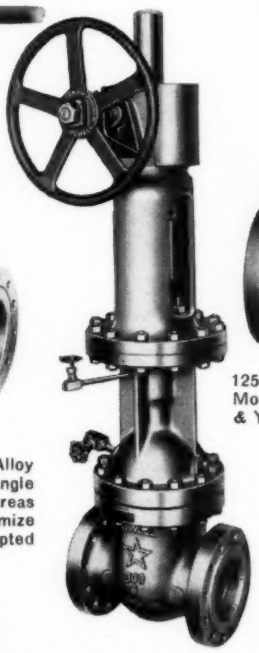
Class 300-pound Cast Alloy Steel Gate Valve, built to handle high temperatures. Has 12" port size venturied to 20" size end flanges to accommodate insulated pipe. Has explosion-proof electric motor operator.

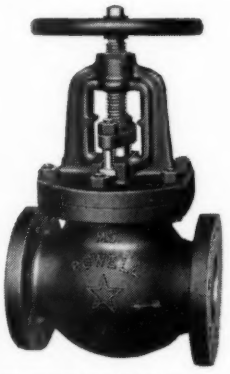
Large 125-pound Iron Body Bronze Mounted or All Iron O. S. & Y. Gate Valve.

125-pound Iron Body Bronze Mounted or All Iron "Master Pilot" Gate Valve.

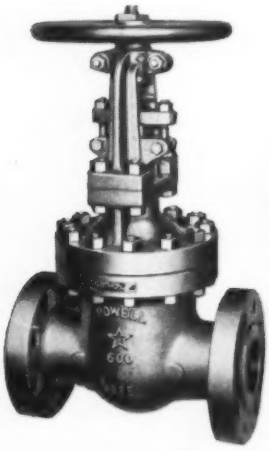


Class 300-pound Cast Alloy Steel O. S. & Y. Angle Valve. Streamlined areas through the body minimize pressure drop. Adapted for high temperatures.

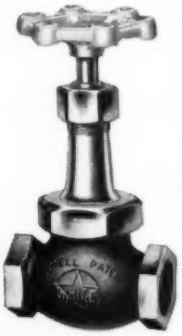




125-pound Iron Body Bronze Mounted or All Iron O. S. & Y. Globe Valve.

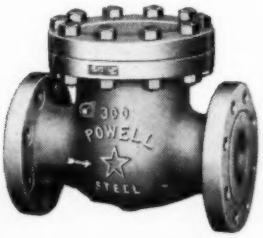


Class 600-pound Cast Steel O. S. & Y. Gate Valve.

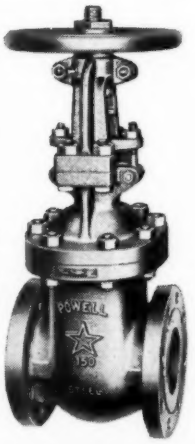


200-pound Bronze Globe Valve with renewable stainless steel seat and regrindable, renewable, wear-resisting "Powellium" nickel-bronze disc.

200-pound Bronze Gate Valve with renewable "Powellium" nickel-bronze disc.



Class 300-pound Cast Steel Swing Check Valve.



Class 150-pound Cast Steel O. S. & Y. Gate Valve.

Class 300-pound Cast Alloy Steel Gate Valve for ultra-high temperatures as in T. C. C. Has drain valves in stuffing box and bonnet and cooling fins to dissipate the heat. Gear operated.

POWELL VALVES

TABLE 1—West Texas-New Mexico Crude Processing Yields and Sulfur Content

	Case 1		Case 2		Case 3	
	Barrels	Per Cent by Weight of Sulfur	Barrels	Per Cent by Weight of Sulfur	Barrels	Per Cent by Weight of Sulfur
Crude fractionation						
Crude-oil charge	100.0	1.81	....	....	100.0	1.81
10-lb naphtha	21.1	0.21	....	....	21.1	0.21
Reformer stock	12.5	0.56	....	....	9.0	0.51
Furnace oil	23.5	1.23	....	....	10.0	0.81
Gas oil	5.0	1.80	....	....	22.0	1.46
Reduced crude	38.0	3.02	....	....	38.0	3.02
Viscosity breaking						
Reduced-crude charge	38.0	3.02	....	....	38.0	3.02
10-lb gasoline	3.6	1.0	....	....	3.6	1.0
Light gas oil	13.2	1.60	....	....	13.2	1.60
Evaporated tar	21.4	3.43	....	....	21.4	3.43
Vacuum flashing						
Evaporated-tar charge	....	....	....	....	21.4	3.43
Heavy gas oil	....	....	....	....	8.0	2.30
Fuel oil	....	....	....	....	13.4	4.04
Thermal cracking						
Gas-oil charge	18.2	1.67	....	....	....	....
Crude-oil charge	....	....	100.0	1.81	....	....
10-lb gasoline	9.3	0.37	56.3	0.36	....	....
Fuel Oil	7.9	2.80	32.2	3.04	....	....
Reforming						
Reformed-stock charge	12.5	0.56	....	....	9.0	0.51
10-lb gasoline	11.1	0.315	....	....	7.9	0.287
Catalytic cracking						
Gas-oil charge	....	....	....	....	43.2	1.67
10-lb gasoline	....	....	....	....	15.3	0.40
Furnace oil	....	....	....	....	15.8	1.33
Fuel-oil	....	....	....	....	5.2	1.89
Coke (pounds)	....	....	....	..	825	4.55
Naphtha desulfurization						
10-lb naphtha charge	21.1	0.21	....	....	21.1	0.21
10-lb naphtha product	21.1	0.042	....	....	21.1	0.042
Furnace-oil desulfurization						
Furnace-oil charge	23.5	1.23	....	....	10.0	0.81
Furnace-oil product	23.5	1.13	....	....	10.0	0.69
Gas recovery, purification, polymerization, and alkylation						
10-lb polymer and/or alkylation product	1.0	0.10	4.8	0.10	4.7	0.10
Dry gas (pounds)	1093	8.08	2561	23.5	1402	10.6
Unisol treating total thermally-cracked, viscosity-broken, and reformed 10-lb gasoline						
10-lb naphtha charge	24.0	0.437	56.3	0.36	11.5	0.502
10-lb naphtha product	24.0	0.357	56.3	0.25	11.5	0.410
Acid treating heavy fraction, Unisol treating light fraction of total thermally-cracked, viscosity-broken, and reformed 10-lb gasoline						
Acid treating:						
Heavy naphtha charge	21.2	0.425	49.5	0.36	10.2	0.478
Heavy naphtha product	21.0	0.290	49.0	0.25	10.1	0.326
Unisol treating:						
Light naphtha charge	2.8	0.543	6.8	0.33	1.3	0.739
Light naphtha product	2.8	0.122	6.8	0.07	1.3	0.176

oil boiling-range material by vapor-phase clay treating is possible, but frequent regeneration of the clay is required. Fig. 20 contains laboratory regenerative desulfurization data for a 0.93% sulfur-content West Texas straight-run furnace oil. From this plot it is apparent that the extent of desulfurization is a function of the coke deposit on the catalyst.

Overall Process Summary

To summarize the data presented thus far, three of the many possible methods of completely processing West Texas crude to fuel products are shown in accompanying Fig. 21, 22, and 23. Volume and sulfur-content data for the charge and products in the various operations are shown in Table 1. Yield, octane-number, and sulfur-content data for the final products in the various cases are summarized in Tables 2, 3, and 4. It should be emphasized that no attempt has been made to achieve a direct comparison of catalytic vs.

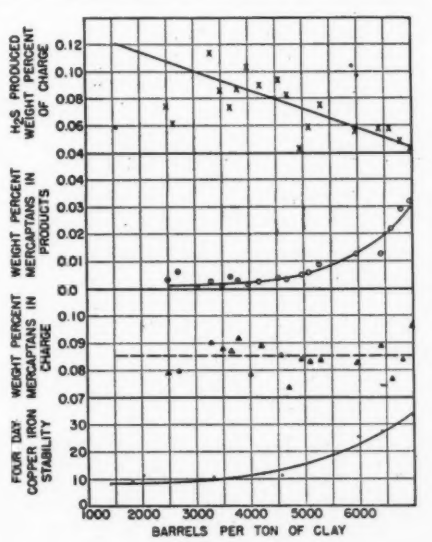

H2S PRODUCED WEIGHT PERCENT OF CHARGE
WEIGHT PERCENT MERCAPTANS IN PRODUCTS
WEIGHT PERCENT MERCAPTANS IN CHARGE
FOUR DAY COPPER IRON STABILITY
BARRELS PER TON OF CLAY

Fig. 19—Catalytic desulfurization of West Texas furnace oil

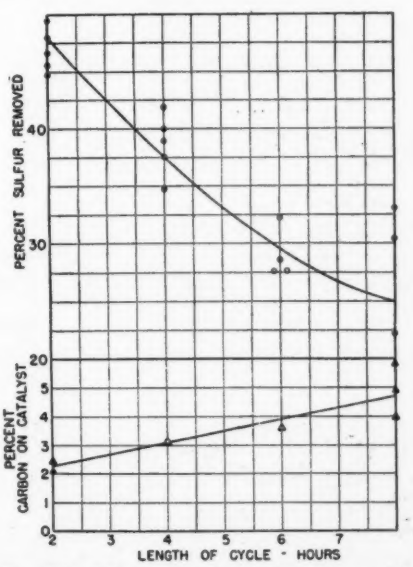


Fig. 20—Effect of carbon deposit on sulfur removal in the catalytic desulfurization of West Texas furnace oil

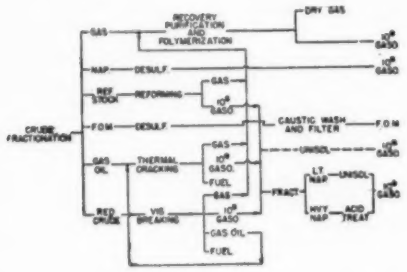


Fig. 21—Case 1 method for processing West Texas crude to fuel products

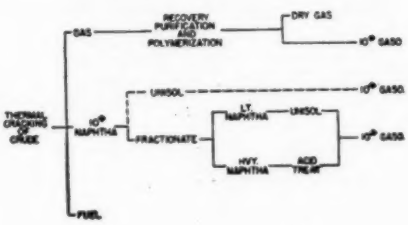


Fig. 22—Thermal cracking of West Texas crude (Case 2)

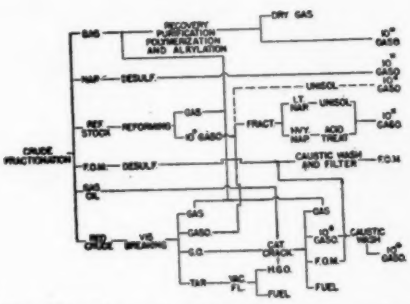


Fig. 23—Case 3 method for processing West Texas crude

thermal cracking, and that many other variations are possible in Cases 1 and 3.

Examining the product summaries, it can be seen that the gasoline produced from West Texas crude varies from 0.15 to 0.25% sulfur content, depending to a great extent on the relative yields of gasoline, furnace oil, and residual fuel oil produced. Further, it should be noted that, whereas Unisol treating does not produce as low a sulfur content as the most costly acid-treating operation, the Unisol-treated product is fully competitive from an octane-number value standpoint.

The final choice of the exact processing scheme employed will depend on many factors, but it is hoped that the data presented will narrow the field for those refiners to whom sour crudes are exactly that. For each individual refinery, the ratio of sweet to sour crude and the ratio of motor fuel to distillate and residual fuel oils will pretty definitely set the complexity of processing required to meet the competitive octane-number, sulfur, and other performance specifications.

Acknowledgement

The help of J. H. McCullough, B. E. Milner, G. P. Masologites, and W. A. Saylor in preparing the charts and correlations used in this report is gratefully acknowledged.

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TABLE 2—West Texas-New Mexico Crude-Processing, Overall-Product-Yield, Octane-Number, and Sulfur Data

	Case 1					
	Acid-treated			Unisol-treated		
Stream	Volume Barrels Per 100 Bbl. of Crude	CFR Research Octane No. Without Addition of Tetraethyl Lead	Sulfur Per-Cent by Weight	Volume Barrels Per 100 Bbl. of Crude	CFR Research Octane No. Without Addition of Tetraethyl Lead	Sulfur Per Cent by Weight
Desulfurized straight-run naphtha	21.1	61.1	0.042	21.1	61.1	0.042
Reformed gasoline	11.0	66.4		11.1	67.8	
Viscosity-broken gasoline	3.6	70.9	0.273	3.6	72.3	0.357
Thermally-cracked gasoline	9.2	76.8		9.3	78.2	
Polymer or alkylate	1.0	110	0.10	1.0	110	0.10
Total 10-lb gasoline	45.9	Without TEL ..... 67.4 1 ml TEL per gal. .. 76.1 With 2 ml TEL per gal. 79.6	0.165	46.1	Without TEL ..... 68.1 1 ml TEL per gal. .. 76.5 With 2 ml TEL per gal. 79.9	0.210
Furnace oil	23.5	....	1.13	....	....	....
Fuel oil	29.3	....	3.28	....	....	....

TABLE 3—West Texas-New Mexico Crude-Processing, Overall-Product-Yield, Octane-Number, and Sulfur Data

	Case 2					
	Acid-treated			Unisol-treated		
Stream	Volume Barrels Per 100 Bbl. of Crude	CFR Research Octane No. Without Addition of Tetraethyl Lead	Sulfur Per Cent by Weight	Volume Barrels Per 100 Bbl. of Crude	CFR Research Octane No. Without Addition of Tetraethyl Lead	Sulfur Per Cent by Weight
Desulfurized gasoline	55.8	68.4	0.230	56.3	69.8	0.250
Polymer or alkylate	4.8	110	0.10	4.8	110	0.10
Total 10-lb. gasoline	60.6	Without TEL ..... 71.7 1 ml TEL per gal. .. 79.6 With 2 ml TEL per gal. 82.8	0.220	61.1	Without TEL ..... 73.0 1 ml TEL per gal. .. 80.4 With 2 ml TEL per gal. 83.7	0.239
Fuel oil	32.2	....	3.04	....	....	....

TABLE 4—West Texas-New Mexico Crude-Processing, Overall-Product-Yield, Octane-Number, and Sulfur Data

	Case 3					
	Acid-treated			Unisol-treated		
Stream	Volume Barrels Per 100 Bbl. of Crude	CFR Research Octane No. Without Addition of Tetraethyl Lead	Sulfur Per Cent by Weight	Volume Barrels Per 100 Bbl. of Crude	CFR Research Octane No. Without Addition of Tetraethyl Lead	Sulfur Per Cent by Weight
Desulfurized straight-run naphtha	21.1	61.1	0.042	21.1	61.1	0.042
Reformed gasoline	7.8	66.9		7.9	68.3	
Viscosity-broken gasoline	3.6	70.9	0.312	3.6	72.3	0.410
Polymer or alkylate	4.7	110	0.10	4.7	110	0.10
Catalytically-cracked gasoline	15.3	94.3*	0.40	15.3	94.3*	0.40
Total 10-lb. gasoline	52.5	Without TEL ..... 79.2 1 ml TEL per gal. .. 85.3 With 2 ml TEL per gal. 87.8	0.214	52.6	Without TEL ..... 79.5 1 ml TEL per gal. .. 85.4 With 2 ml TEL per gal. 87.8	0.235
Furnace oil	25.8	....	1.14	....	....	....
Fuel oil	18.6	....	2.97	....	....	....

* Base octane number of catalytically-cracked gasoline: pool octane number is corrected for increased blend octane number of catalytically-cracked gasoline.

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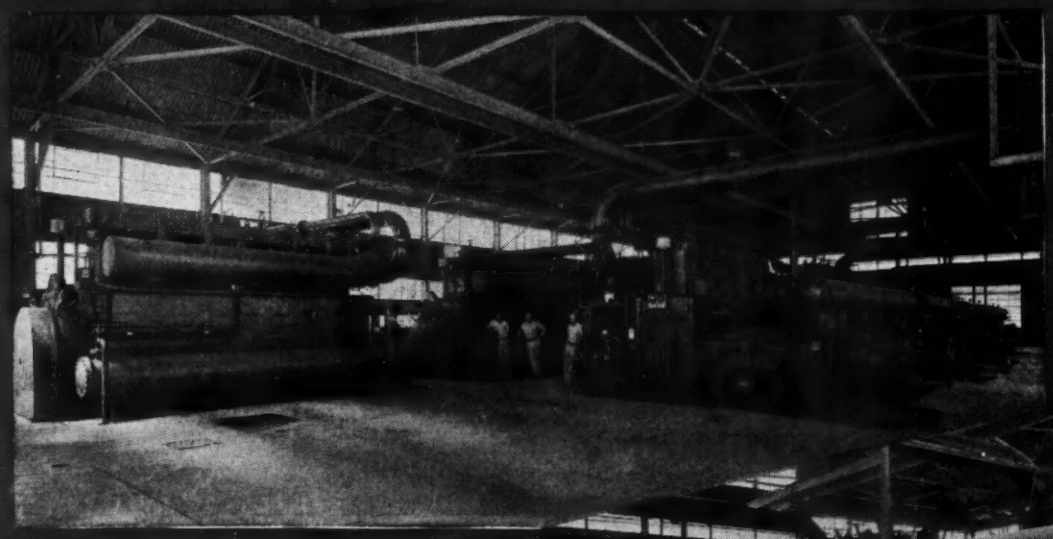

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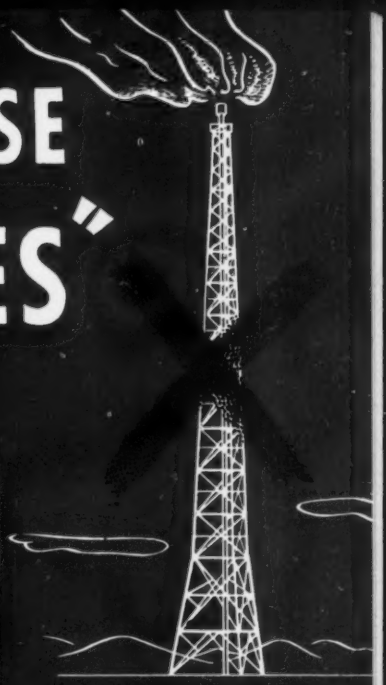

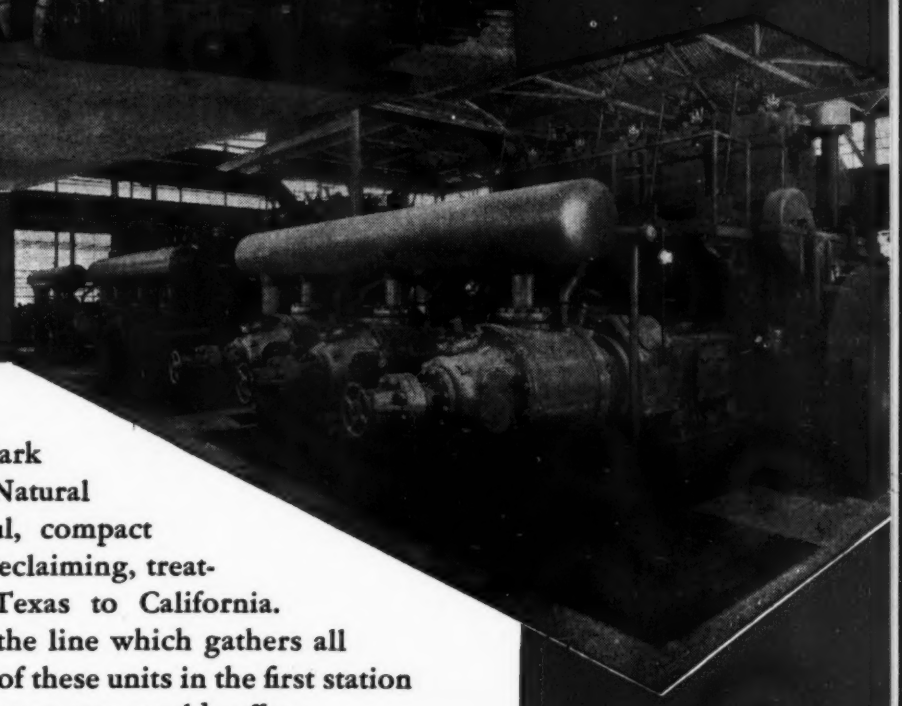

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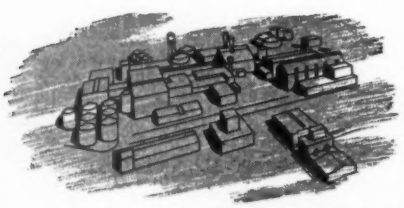

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Tests on Cracking Catalysts Used In Control of Commercial Units

PAPERS

A variety of test methods, developed by oil and allied companies, for evaluating cracking catalysts as to their behavior in the catalytic cracking of petroleum, were presented at the Symposium on this subject Nov. 10, sponsored by the Refining Division as part of the API annual meeting in Chicago.

The methods can be briefly summarized as short-time procedures, using small amounts of catalysts measured in grams or milliliters to attain data that can be applied in the control of the large-scale commercial units circulating hundreds of tons of catalyst a day. The tests are also included in general research work to improve the efficiency of commercial catalytic cracking operations.

The 14 test methods presented at the Symposium are of three classifications:

1—Laboratory physical and chemical methods which determine properties of cracking catalysts such as, for example, the aromatic adsorption index, which can be correlated with the activity of the catalyst in a hydrocarbon conversion reaction.

One group of this type of tests includes methods for directly determining particle-size distribution, which is an important factor in plant operation.

2—Laboratory and bench-scale cracking units, where the catalyst is tested under conditions simulating those in commercial operations. Catalyst activity here is based on assembled test data, such as conversion into liquid products, gas- and coke-formation and others. There are various modifications of these methods. For example, out of test work over the past 11 years, in which some 5000 small-scale runs have been made, the Whiting research laboratories of Standard Oil Co. (Indiana) have developed a fixed-bed test unit which passes about 200 ml. of a standard gas oil feed stock through 80 ml. of powdered catalyst. Relative activity is reported as based on the determined activity of a reference catalyst.

Included in this group of tests described at the Symposium was the so-called standard CAT "A" test of the Houdry Process Corp., which at present is being used in more than 20 different laboratories. This test, which consists of a small laboratory-scale cracking unit taking 200 ml. batches of catalysts, was first developed in 1938.

3—One example was presented at the symposium of the third classification of catalyst test methods. This is by pilot-plant test in the same equipment in which general research and development work on catalytic cracking is carried on. Universal Oil Products Co. technologists described their procedure in a pilot plant charging 3-4 barrels per day of feed stock.

Harry Levin, The Texas Co. laboratories, Beacon, N. Y., presided at the morning session of the Symposium and E. L. Baldeschwieler, Esso Laboratories, Standard Oil Development Co., Elizabeth, N. J. at the afternoon session.

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Note: Complete texts of all papers presented in this Symposium will be published in the Proceedings of the Refining Division of the 27th Annual API meeting. They should be available the early part of 1948, Refining Division Director W. T. Gunn has announced.

Accurate Particle-Size Determination of Fluid Catalyst by Coordination of Roller Analysis and Microscopic Examination

By John T. Wiley, Joe E. Deloney, and S. Walter Denton, The Texas Co., Port Arthur, Texas

AN increasing demand for an accurate method for sub-sieve particle-size distribution in cracking catalysts, because of appreciation of its chemical and physical importance, has made it necessary to evaluate different methods that are used to determine the particles lying within different ranges, this paper brings out.

Experience with the Roller method has brought out modifications that have tended to improve its accuracy with fluid catalyst samples. The accuracy and reproducibility of the Roller apparatus is described; some of the variables are discussed and the solution of the variables is given. The Roller physical method for determining particle-size distribution of sub-sieve catalyst is described as follows:

"The Roller method is an air-elutriation method used to separate materials into fractions of different size particles by carrying upward in an air stream those particles too small to settle against the upward velocity of the air. Elutriation methods are based on the same laws, and are affected by the same conditions, as sedimentation methods.

"One advantage of elutriation methods over sedimentation methods is that elutriation methods yield fractionated products which may be studied separately from the whole material, and photomicrographs can be made of each separation for permanent records if desired.

"The theory or law upon which the Roller design depends is Stokes law, as follows:

$$v = \frac{10^{-8}\, g p d^2}{18n}$$

Where:

- v = terminal velocity (cm per sec) in stationary fluid.
- g = constant of gravitation [in cgs (metric units)].
- p = density of particle, g per cu cm.
- n = viscosity of fluid [in cgs (metric units)].
- d = diameter of sphere in microns (1 micron = 10^{-4} cm)."

The Roller method of analysis as used in The Texas Co.'s Port Arthur laboratories is described in detail, including the examination of separations under a measuring microscope to determine the homogeneity and quality of the separation. Reproducibility is indicated to be approximately ± 1.3% from the average, due to improved apparatus and strict adherence to standardized procedure. The precautions essential to accurate reproducible results are listed. The conclusion of the paper states:

"The Roller method with carefully standardized apparatus, using the modified filter trap and close correlation of air flow with density, is a convenient rapid method that gives reproducible results when used along with a measuring microscope. Photomicrographs can also be taken on any separation for a permanent record, if desired. An experienced operator can make 4 Roller analyses, and check the results under the microscope, within 8 hours."

Aromatic Adsorption Index for The Estimation of Surface Area And Catalytic Activity

By T. W. Pratt, M. W. Kellogg Co., New York City.

WHILE no single index to all of the catalyst variables is available, the aromatic adsorption index provides a means for rapidly and simply detecting changes in the catalytic surface available, and changes in activity due to the destruction of catalytic surface either by heat or steam.

Detailed procedure for the determination of the aromatic adsorption index, as well as experimental data and correlations concerning the effect of variables upon the resulting value of the index, are given in an appendix to the paper, which describes briefly the steps in determination of the index as follows:

1. Drying—2 hours at 850° F. for fresh catalyst, or 2 hours at 1000° F. for used catalyst.

2. Adsorption — 3.50 g. of dried catalyst are shaken with 5.0 ml. of a 30% toluene—70% isooctane mixture for 2 hours at 77° F.

3. Analysis—The analysis of the solution before and after contact is performed by determining the refractive index n_D. The change in the refraction index times 10^4 is the adsorption index.

The aromatic adsorption index affords a means of estimation of the "nitrogen area" of the catalyst, regardless of the catalyst source or history. "Butane areas" can be estimated accurately from the index if the history of the catalyst sample is known, inasmuch as it has been found that the relationship is altered depending upon whether the surface has been subjected to heat or steam.

Correlations of the aromatic adsorption index with activity for type "A" and other synthetic catalysts show the estimated activity of a given sample can be expected to be within ±2% (conversion) of the true activity. In most cases correlations for catalysts from the same source used in different units under different conditions will be sufficiently accurate for the estimation of activity. However, it is usually possible to obtain superior correlations for data taken from a single unit.

"The simplicity and rapidity of the determination make it an easy matter for any refiner to follow catalyst changes at very frequent intervals," states the conclusion of the paper. "Very little equipment is required, nor is highly skilled personnel necessary. The determination is not empirical as regards time of contact. The system reaches equilibrium in less than 2 hours, and may remain indefinitely without affecting the results. Furthermore, there is no problem in obtaining the reagents in the pure state, nor do they deteriorate from contact with the atmosphere."

The Aromatic Adsorption Index as a Rapid Method For Approximating Catalyst Activity

By W. W. Scheumann and A. R. Rescorla, Cities Service Refining Corp., Lake Charles, Louisiana.

IN the laboratories of Cities Service Refining Corp. at Lake Charles, the standard procedure used for activity determinations for catalysts used in fluid catalytic cracking operations has been the Kellogg fluidized fixed-bed activity test. However, for a rapid test method or for a check of results by the standard method, the aromatic adsorption index has also been used. Data is presented showing the degree of correlation between the two methods.

This physical method as used in the Cities Service laboratory is a modification of a procedure developed by the Petroleum Research Division of M. W. Kellogg Co. It is based on the ability of a cracking catalyst to adsorb selectively aromatic hydrocarbons from a hydrocarbon mixture. A brief description of the method is as follows, as given in the paper, which also includes a detailed description of the Cities Service modification:

"The test is carried out by first heat treating the catalyst sample at a temperature of 850° F. A 3½-g. sample is then weighed into an adsorption vessel to which is added 5 ml. of a hydrocarbon mixture consisting of 30% toluene and 70% isooctane. The adsorption vessel is shaken in a mechanical shaker for 2 hours, after which the refractive

index is determined on the supernatant liquid.

"The aromatic adsorption index is the difference between the refractive index of the original hydrocarbon mixture and that of the hydrocarbon mixture after contact with the catalyst multiplied by 10^4.

"This determination requires approximately 5 hours' elapsed time, and it is quite apparent that a single laboratory operator can carry out a considerable number of such determinations simultaneously, so that the man-hour requirements per determination are quite small. In addition, the only piece of expensive laboratory equipment required is a precision refractometer equipped for constant-temperature work."

Prediction of the Activity Of Cracking Catalysts From Heat of Wetting

By Ivor W. Mills, Sun Oil Co., Marcus Hook, Penna.

STUDY was made of physical properties of catalysts which might be used for correlation purposes, the basic activity used for correlation being the volume per cent of motor gasoline produced as in the Alexander-Shimp technique (described in a later paper). A physical method was developed using a heat-of-wetting correlation. The catalyst types investigated were the pelleted clay type and the synthetic silica-alumina type, both in the bead and pelleted form.

"Concerning the properties of these catalysts which might be useful for correlation with activity," states the paper, "it is noticed they are all adsorbents and that they exhibit high heats of adsorption.

"In an attempt to develop a correlation, both properties were investigated. The amount of heat evolved when a catalyst is contacted with a liquid is referred to as 'heat of wetting.' This property was determined for a series of catalysts using liquids including methanol, water, acetone, and acetic acid. Of these liquids, methanol appeared to be the most favorable, as it gave a good temperature rise, could be obtained in a pure state, and was easy to handle. Therefore, heat-of-wetting data for the rest of the paper imply the use of methanol.

"The same series of catalysts was used to investigate adsorption characteristics. In these experiments, 10g of powdered catalyst were contacted with 15 ml. of a 50-50%-by-volume mixture of methanol and benzene. The mixture was placed in a shaking machine until the refractive index of the liquid phase reached a constant value. The refractive-index change is a measure of the amount of methanol selectively adsorbed. This liquid pair is probably not ideal for studying adsorption characteristics, but it was sufficiently good to show that heat-of-wetting and adsorption capacity are directly related."

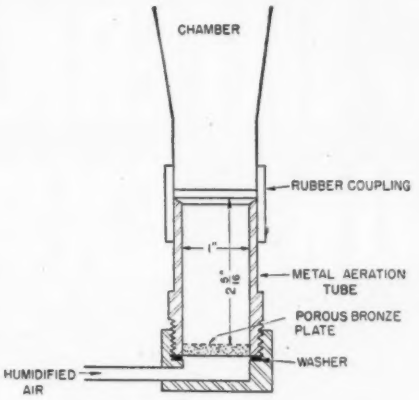

Fig. 1—Modified Roller catalyst aeration tube

The apparatus used in the heat-of-wetting test is described, the test procedure and results of studies, including study of certain conditions which may complicate the correlation, such as water present on a catalyst and carbon and iron oxide contaminants The effect on the correlation of blends of catalysts of different activities is discussed.

Modification in the Roller Analysis for Determination of Particle-Size Distribution

By George L. Matheson, Standard Oil Development Co., Elizabeth, N. J.

WORK with the Roller physical method of analysis for determination of particle-size distribution of cracking catalysts in the Standard Oil Development Co. laboratories included study of the problem of particle fragmentation during analysis and also dealt with the effect of changing relative humidity of the elutriation air.

In the determination of particle-size distribution of powdered catalyst, it is noted, it is important to minimize particle-size alteration due to attrition or fragmentation. The Roller U-tub and air jet is potentially a grinder with its jet velocity of 220 ft./sec. (when passing 10 grams/min. of air through the 0.070-in. jet) and impinging air. Whether this grinding will affect the particle size depends on the catalyst's resistance to attrition and fragmentation.

To test this property a modified aeration tube (Fig. 1) was made, provided with an air distributor in the form of a porous plate filled with a multitude of tortuous openings that prevent catalyst from falling through, and also will distribute the air evenly across the surface.

The modified apparatus was found to give good results with all fluidizable catalysts except for a few samples containing 90% or more in the 0- to 10- micron range. The difficulty here was due to the tenacity with which this sample clings to the side walls. For such samples, the standard Roller apparatus with the air jet was found preferable, although it did not entirely eliminate the difficulty.

Comparative tests were carried out to study the effect of relative humidity of the elutriation air on the determination of particle-size distribution. The modified aeration tube was used. The results indicated, it is pointed out, that a high relative humidity should be used for Roller analyses. The reason for the difference in the behavior of new and regenerated catalysts, referring to certain tests, is described as probably due to the new catalyst being a much better conductor of electricity than regenerated catalyst.

The results of the test work indicate, the paper states, that 50 to 70% relative humidity should be used in Roller analyses.

"The performance of the Roller apparatus should be checked occasionally, particularly when running new kinds of powders, for both quality of separation and size range of cuts," it was stated. For this purpose the microscope with its micrometer eye piece is indispensable for examining the fractions obtained."

Plant Control Tests for Particle-Size Distribution Of Fluid Cracking Catalysts

By K. D. Ashley and W. B. Innes, American Cyanamid Co., Stamford, Conn.

THE manufacture of large quantities of fluid catalyst necessitated the development of rapid and convenient control methods for determining the particle-size distribution in order to meet customer specifications.

In order to select the best methods available, study was made of the various physical tests in use for determining particle-size distribution. Methods examined included: Sedimentation (a, Incremental; b, Cumulative; c, Wagner's turbidimeter methods; d, elutriation): Screening: Microscopic examination.

The conclusion was drawn that the methods commonly in use on cracking catalysts for the subsieve fractions (passing a 200-mesh sieve) should show satisfactory agreement provided conditions are such that Stokes law is obeyed. The commonly used methods are the hydrometer in-

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cremental sedimentation test, Roller air elutriation, and the sedimentation balance method.

Since the subsieve methods studied appeared to be equally reliable, the most rapid method, the hydrometer method, was chosen for plant testing. Details are given as to how this method of test was adapted to testing cracking catalyst. The conclusion of the paper states:

"For the subsieve particles, the sedimentation methods should all yield reliable results, if a 'non-aggregating fluid' is used, and if the proper densities are employed in Stokes equation and for calculating the 'per cent finer.' *Iso*propyl alcohol was the most suitable liquid found from the standpoint of not causing aggregation, and because it could be purchased cheaply in pure form. For a porous substance, the particle as defined by Stokes equation is that with pores filled with the sedimenting liquid. Hence, the pore-filled density should be used in its application.

"For ground catalyst, the average particle density in *iso*propyl alcohol is 1.51, and for microspheroidal catalyst it is 1.40. These values increase for both catalysts after use in cracking units. A skeletal density of 2.30 is used in the 'per cent finer' equation.

"The hydrometer method is a very satisfactory method for determining the particle-size distribution of the portion of the sample passing a 200-mesh sieve. The results by this method check very closely with those obtained by microscopic count, etc."

The research work of the American Cyanamid Co. determined that screening offered the quickest and most accurate method for determining particle-size distribution for catalysts above 62 to 74 microns (230- to 200-mesh sieves). Blinding becomes too troublesome with finer screens. The screen method used by the company is described.

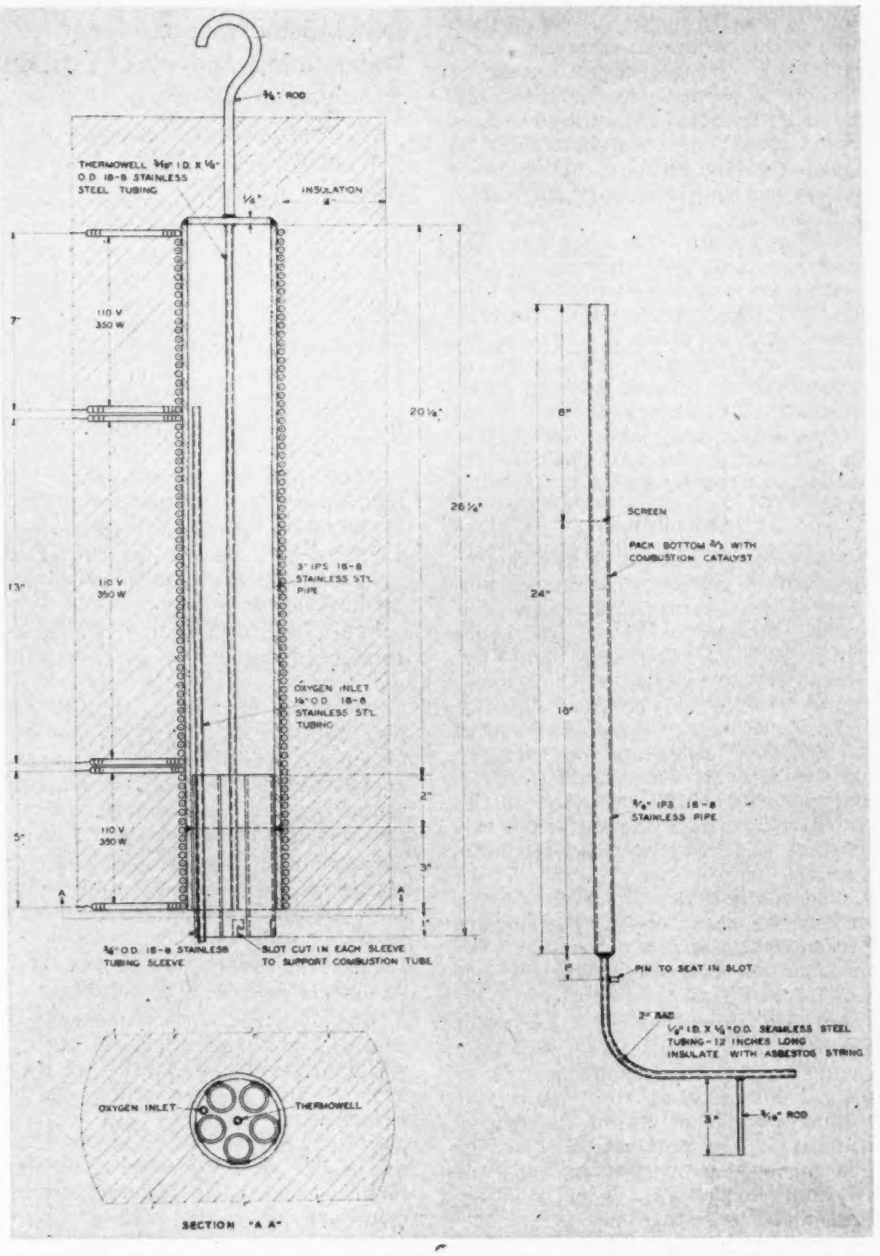
⅛" ROD
THERMOWELL ¾" I.D. X ⅛" O.D. 18-8 STAINLESS STEEL TUBING
INSULATION
110 V 350 W
110 V 350 W
110 V 350 W
3" IPS 18-8 STAINLESS STL PIPE
OXYGEN INLET ¼" O.D. 18-8 STAINLESS STL TUBING
¾" O.D. 18-8 STAINLESS TUBING SLEEVE
SLOT CUT IN EACH SLEEVE TO SUPPORT COMBUSTION TUBE
SCREEN
PACK BOTTOM ⅔ WITH COMBUSTION CATALYST
¾" IPS 18-8 STAINLESS PIPE
PIN TO SEAT IN SLOT
2" RAD
¼" I.D. X ½" O.D. SEAMLESS STEEL TUBING - 12 INCHES LONG INSULATE WITH ASBESTOS STRING
⅛" ROD
OXYGEN INLET
THERMOWELL
SECTION "A A"

Fig. 2—Electrical furnace and combustion tube used in cracking catalysts tests to determine the carbon content of catalysts

Simplified Method for Determination of Carbonaceous Deposits on Cracking Catalysts

By F. E. Ray and A. E. Potas, Socony-Vacuum Research Laboratories, Paulsboro, N. J.

A SIMPLE method has been devised for determining the carbon content of catalysts, for use in controlling continuous catalytic cracking operations. The method, which is applicable directly to granular catalysts coarser than 60-mesh, has the following advantages:

1—It is unnecessary to grind the catalyst to a fine powder.

2—The equipment required is simple and inexpensive.

3—Five determinations can be made simultaneously with a total elapsed time of 2 hours and the expenditure of 1 man-hour.

4—It does not require a technically trained operator to conduct the test.

The standard deviation of the test method is said to be ±3% of the total carbon present in the range of 0 to 7% by weight on the catalyst. The test apparatus, procedure and calculations are described as follows:

Apparatus

The apparatus shown in Fig. 2 consists of an electrically heated furnace, combustion tubes, and a gas-collecting system and is fully described in the paper.

Auxiliary equipment consists of a flow meter for the control of the oxygen supply, an Orsat apparatus, a semi-analytical balance, a riffle sampler, and a potentiometer for measurement of the furnace temperature.

Test procedure is described as follows:

"The catalyst sample is carefully riffled to approximately 20 g., weighed to the nearest 0.1 g., and charged to the top section of the combustion tube. Oxygen is passed into the furnace from a cylinder at a rate of 75 ml. per minute for each combustion tube in service. The combustion tube is inserted in the furnace and connected to one of the calibrated gas bottles. The associated reservoir is then lowered to start the flow of brine through the controlling orifice and, hence, the

flow of gas through the combustion tube to the calibrated receiver.

"If the carbon-dioxide content of the gas is greater than 60 per cent, the results should be discarded, a fresh sample of catalyst should be charged to the furnace, and 8 liters of flue gas collected in 2 gas bottles in 180 min.

"After about 4 liters of flue gas have been collected, the gas receiver is shut off and the combustion tube removed from the furnace. The volume of the collected flue gas is recorded, and the gas is analyzed for carbon-dioxide content with an Orsat apparatus. A sufficient number of gas analyses are made so that a majority of the values check to 0.2 per cent of the total gas.

Calculations

"The carbon content of the sample analyzed is calculated from the volume (V in liters) of flue gas collected, carbon-dioxide content (per cent CO_2) of this gas, and the weight (W in grams) of the sample charged to the combustion tube. The volume of flue gas should be corrected to standard conditions of pressure and temperature (760 mm and 0° C), and for the partial pressure of the water vapor from the saturated salt solution at the prevailing temperature."

The method developed for determining the above correction factor is presented in the paper, together with an equation for calculating the per cent of carbon.

In accordance with statistical methods, sufficient determinations should be made on each catalyst so that a majority of the values fall within a range of 6% of the carbon content of the catalyst samples. In general, only two determinations are required to obtain a satisfactory agreement, it is stated.

As a result of test work it was determined that the standard deviation of calculated results from actual determinations is 2.3% of the carbon on the catalyst charged.

Bench-Scale Method for Determining Activity of Cracking Catalysts in Powdered Form

By Hubert McReynolds, The Texas Co., Port Arthur, Texas.

THE ideal fluid catalyst-activity evaluation method should combine as its chief characteristics the following points, as enumerated in this paper:

"It should be capable of testing fluid catalysts without the necessity for pelleting.

"Operating temperatures and temperature gradients should be capable of being measured accurately.

"The process periods should be long enough for fluctuations about a steady state to average out.

"Sufficient charge stock should pass through the unit to insure ample product for testing.

"The conditions under which the test unit separates products into liquid and gas, such as receiver and knock-back condenser temperatures, must be definite and reproducible.

"Distillation of the liquid product should be done by use of an automatic Hempel column to give a precise measure of conversion.

"A definite pretreatment or conditioning of the catalyst, to put it into a 'standard state' before testing, should be adhered to."

The test method in use at Port Arthur is described as follows:

"Briefly, the method consists of charging a standard gas oil through 400 g of powdered catalyst at about 920° F for 30 min, the resulting liquid being stabilized and fractionated to yield 400° F-end-point gasoline. The activity of the catalyst may be reported in terms of conversion (100 minus per cent by volume gas oil) or the per cent distilled plus loss (D + L) on the total liquid product. This method may be correlated with other evaluation methods, for example, the Jersey method, to express catalyst activity in other terms.

"A schematic flow diagram of the test unit is shown in Fig. 3. It will be noted that it consists of a charge system, an upflow fluidized fixed-bed reactor, a brine-cooled condenser, a gas meter, and a gas-accumulating drum. When this equipment is used for activity determinations, a calcined catalyst is loaded into the unit, and a few minutes are allowed for the catalyst to come to temperature.

"The run is then made without any further delay. As soon as the run has been completed, the catalyst is purged with flue gas and drawn from the unit. No provision is made to regenerate or reuse the catalyst; if additional runs are required, they are made on fresh portions of the same catalyst."

Test procedure is described in detail. Data obtained in the test may be expressed in two ways, to estimate catalyst activity.

"1) Per cent conversion, which is 100 minus per cent by volume of gas-oil yield; basis, fresh feed; i.e.: gasoline, gas and carbon plus loss. Fresh Diakel catalyst results in a conversion of about 55% on this basis.

"2) The Texas Co. D+L, which is 100 minus per cent by volume of gas-oil yield; basis, total liquid product; i.e.: 400° F.-end-point gasoline produced from the total liquid plus distillation loss. Fresh Diakel catalyst has an activity of approximately 40% on this basis."

The reproducibility of catalyst evaluation data both on the basis of Texas D+L and conversion is shown.

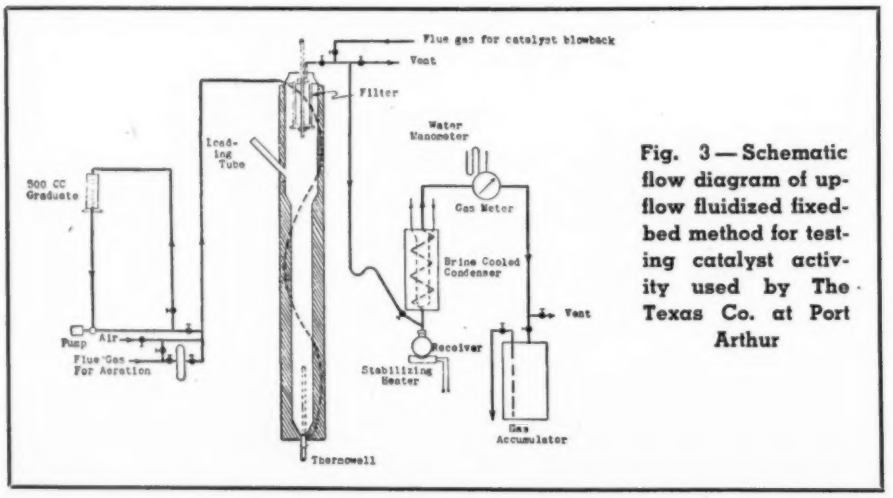

Fig. 3 — Schematic flow diagram of upflow fluidized fixed-bed method for testing catalyst activity used by The Texas Co. at Port Arthur

Design and Operation of a Bench-Scale Automatic Catalyst-Aging Unit

By T. Rice. Gulf Research & Development Co., Pittsburgh, Penna., and F. E. Ivey, Jr., Gulf Oil Corp., Philadelphia, Penna.

AS AN AID in evaluating commercial and experimental catalysts for catalytic cracking processes, a bench-scale unit has been designed and is being operated for investigating the aging characteristics of pelleted catalysts.

The unit, with slight modifications, can also be used to determine the product distribution obtainable from catalysts at various activity levels, the effect of operating variables on product distribution and other information pertaining to the operation of commercial catalytic cracking equipment.

The two primary considerations when the catalyst-aging unit was de-

signed were: 1) comparability of the operation with commercial and pilot-plant operation; 2) ease and simplicity of operation with a minimum of attention.

The first of these objectives was accomplished by patterning the design of the bench-scale unit after a fixed-bed pilot plant which was not suitable for extensive aging studies, due to its size. It appeared that ease of operation with a minimum of manpower could be attained only by the use of automatic equipment controlled by a cycle timer. The test equipment and its operation is described briefly as follows in the paper, which also includes a diagram of the equipment and full details of its operation:

"The unit was designed to operate on a 30-min-cycle with an on-stream period of 10 min., a vacuum- and nitrogen-purge period of 5 min., a regeneration period of 10 min. and a nitrogen-purge period of 5 min. The cycle timer was used to control the flow by opening and closing in sequence solenoid or motor-operated valves, and by starting and stopping the motor on the oil charge pump. The timer was also used in conjunction with safety features for shutting down the unit in case of equipment failure.

"The principal parts of the unit include a feed tank, a charge pump, a combined preheater and reactor, a product condenser and receiver, a gas meter, a vacuum purge condenser and receiver, a vacuum pump, and a flue-gas combustion and absorption train, in addition to the equipment for automatic control.

"The feed tank, product receiver, and vacuum purge receiver have capacities sufficient to operate the unit for about 36 hours without attention."

Typical catalyst aging data is shown in which the activities of the pelletted catalysts were determined by the CAT "A" (Houdry Process Corp. standard laboratory) method before aging and after approximately 350, 700 and 900 cycles of the tests.

Other problems related to catalytic cracking which may be investigated in this unit on a relative basis are:

1—Determination of the cracking characteristics of catalysts at various activity levels or degrees of contamination.

2—Effect of operating variables on product distribution.

3—Effect of various charge stocks on the aging characteristics and contamination of catalysts.

4—Determination of the product distribution from miscellaneous charge stocks.

A Bench-Scale Test Method for Evaluating Cracking Catalysts

By E. R. Birkhimer, S. J. Macuga, and L. N. Leum, Atlantic Refining Co., Philadelphia, Penna.

A SHORT-TIME bench-scale test method has been developed in the research laboratories of this company which has been employed for the past three years in catalyst-control testing for commercial and pilot-plant fluid catalytic cracking units. The test has been found satisfactory for evaluating the activity of a catalyst and has shown considerable promise as a method suitable for measuring product yields.

The test was designed to run under conditions which are within the range used in the refinery when operating with equilibrium catalyst, in order that test conversions, gas yields and coke on catalyst would be on the same order of magnitude as those experienced in the large unit.

Conditions employed in this activity test are given in Table 1. The variables, time on stream, catalyst-to-oil ratio, space velocity and temperature were the best estimates made at the time of probable operating conditions in the plant. Because of the difficulties of operating a short-time, bench-scale test under pressures such as are experienced in commercial units, atmospheric pressure was selected on the basis of convenience.

TABLE 1—Test Conditions—Atlantic Activity Test

Time on stream	12 min
Weight of catalyst-to-oil ratio	10.0 g
Weight of space velocity	0.5 g
Temperature	900° F
Pressure	Atmospheric
Feed rate	2.0 ml per minute
Feed charged per test	24.0 ml (20.0 g)
Catalyst weight	200 g
Pellet size	$\frac{3}{16}$ in. in diameter $\frac{1}{16}$ in. in height
Trap temperatures:	
No. 1 trap	75° F
No. 2 trap	−45 + 1° F
Charge stock	East Texas furnace-oil medium

In order to operate at the desired catalyst-to-oil ratio and space velocity, and at the same time to process sufficient material in each test for an ASTM distillation of the product, each test would have required some 2000 g. of pelleted catalyst. This was objectionable because of the troublesome and time-consuming job of pelleting fluid catalyst and because the test was to be used for pilot-plant and research samples where large quantities of catalyst often are not available.

It was therefore decided to limit the amount of catalyst to 200 g. and to develop the test procedure along lines of handling small amounts of oil. The Atlantic activity test processes 24 ml. of oil per test. To handle this small quantity of oil with a reasonable degree of reproducibility, it was found necessary to use special recovery systems to minimize losses and to develop a distillation technique that could handle as low as 5 ml. of synthetic crude. These developments are described in the paper.

A fixed, pelleted bed of catalyst is used in the test. The standard feed stock is a furnace-oil medium frac-

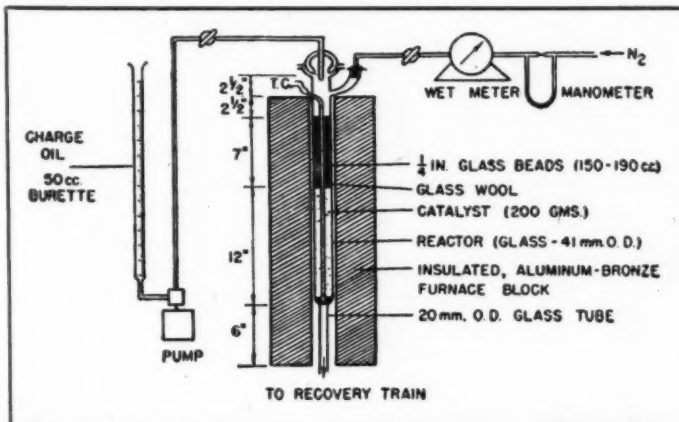

Fig. 4 (above)—Cracking apparatus used in Atlantic Refining Co. activity test

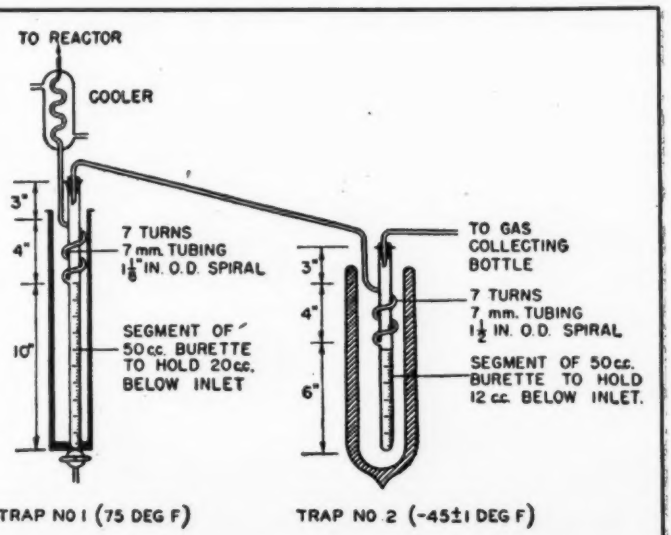

Fig. 5 (right)—Recovery system for the Atlantic test

tion from East Texas crude boiling between 400 and 600° F. and having an API gravity of 37.3. Selection of feed stock was based purely on its availability at the refinery but later work has indicated that low-boiling feed stocks were more sensitive to differences in catalysts than high-boiling feeds.

The cracking apparatus used in the test is shown in Fig. 4. The special recovery system developed to use with the small amount of oil processed in the test, consisting of a water cooler followed by a collection train consisting of two traps is shown in Fig. 5. The distillation apparatus provides for distilling 5 to 10 ml. of liquid.

Calculations made as a routine part of the activity test include:

Carbon yield, % by weight of feed.

Gas yield, % by weight of feed.

Recycle (material boiling above 400° F.) % by volume of feed.

Conversion (100%-recycle), % by volume of feed.

Volumes of liquid recovered.

D + L, % by volume.

Balance, % by volume.

"The activity results of the test are reported by two numbers, viz., per cent by volume of D + L and conversion. The activity of the catalyst from the viewpoint of effectiveness for cracking as separate from selectivity is best expressed by conversion. Per cent by volume of D + L, and particularly the spread in numbers between D + L and conversion, is an indication of product distribution. In addition to these two numbers, carbon yields are also reported as carbon-producing factors."

Results obtained in this short-time bench-scale test are presented, which indicate that it has a contacting efficiency comparable to that found for the commercial fluid catalytic cracking unit.

Determination of Activity And Selectivity of Cracking Catalysts

By Rodney V. Shankland and George E. Schmitkons, Standard Oil Co. (Indiana) Whiting, Ind.

EXPERIMENTAL work on testing cracking catalysts has been carried on in the research and development laboratories of Standard Oil Co. (Indiana) at Whiting in which some 5000 small-scale runs have been made. Pilot-plant work has included both fixed-bed and moving-bed operation, as well as fluid catalyst operation.

This work has resulted in the development and adoption of a fixed-bed, test-unit method applicable to testing catalyst in any form: viz., granules, pills or powder. Steps have been taken to insure a high degree of precision in the control and measurement of the important operating conditions.

Data obtained by this test enables reporting coke (carbon) and gaseous products yields and activity indices at standard reference conditions. By activity index is meant any of such items as the conversion, gasoline yield, D + L (distillate plus loss) on liquid product, etc., which are known as "activity" in many laboratories. However, at this laboratory the custom is to report the relative activity. This last named factor is defined as follows:

"Relative activity is defined as the weight of reference catalyst which, by comparative runs, gives the same degree of cracking as a fixed weight of the test catalyst. This involves the concept of activity units which are proportional to weight of the catalyst as expressed in the example: 2 lb. of catalyst of activity 50 will accomplish the same degree of cracking as 1 lb. of catalyst of activity 100 at otherwise constant (especially temperature) conditions."

Among selectivity factors, carbon-producing is usually rated in the same test-unit cracking run employed for rating activity. Furthermore, the carbon factor is sometimes of more economic significance than the activity and often appears to undergo capricious changes (frequently traced to poisoning) in commercial unit and pilot plants. The gas factor is also conveniently rated in the same run with activity and carbon factor. Other selectivity factors such as the C_4-to-gasoline ratio and octane number, are usually not rated in routine tests, it is brought out in the paper.

After careful consideration of the experimental data accumulated, the method of determining and reporting catalyst activity in terms of relative weights of reference and test catalyst required to achieve equal conversions at otherwise identical cracking conditions was adopted because it appeared to have the following inherent advantages over other methods:

(a) The activity rating reported as relative activity appeared to be more or less independent of the particular type of apparatus, feed stock and standard operating conditions selected for the test method and to depend almost entirely on the inherent activity of the test catalyst and the inherent activity of the reference catalyst.

(b) Because of the above situation, comparable activity ratings should be attainable by different laboratories without the necessity of rigidly reproducing all details of apparatus, feed stock and operating conditions—provided the same reference catalyst is employed.

(c) It appeared possible to incorporate improvements in the equipment or change details of operating conditions without changing the ratings on the original activity scale.

(d) The interpretation of relative weight activities in terms of their effect on commercial cracking operations should be more direct than ratings reported as primary results of the test.

(e) Relative weight activities should be additive, permitting a ready calculation of the relative activity of a mixture of two or more catalysts.

The downflow fixed-bed method for testing catalyst was adopted as satisfactory for testing powdered catalyst. In addition, granular or pilled catalyst samples can be readily tested in the downflow fixed-bed apparatus.

The reference material used in determining the relative activity of catalysts under test is a very active microspherical silica-alumina catalyst.

The test method as currently used

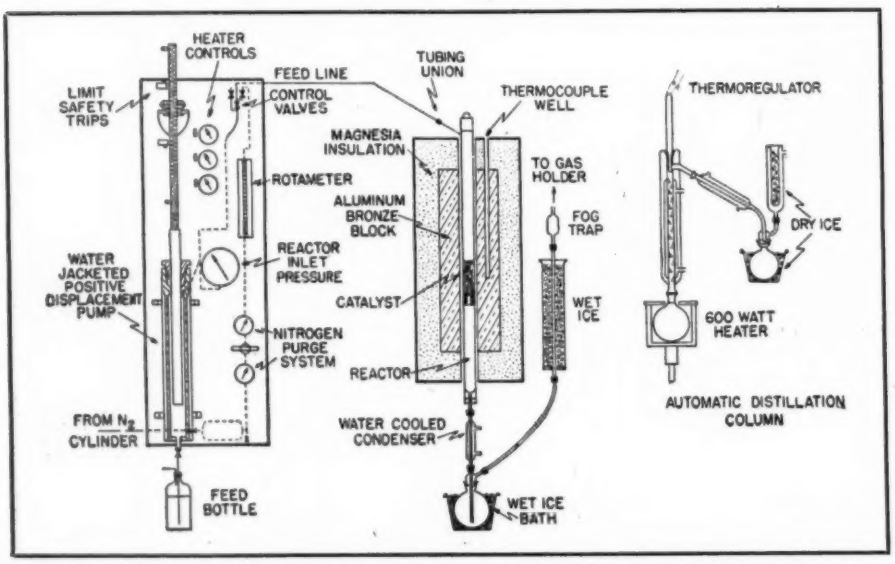

Fig. 6—Diagram of equipment used by Indiana Standard in testing catalysts

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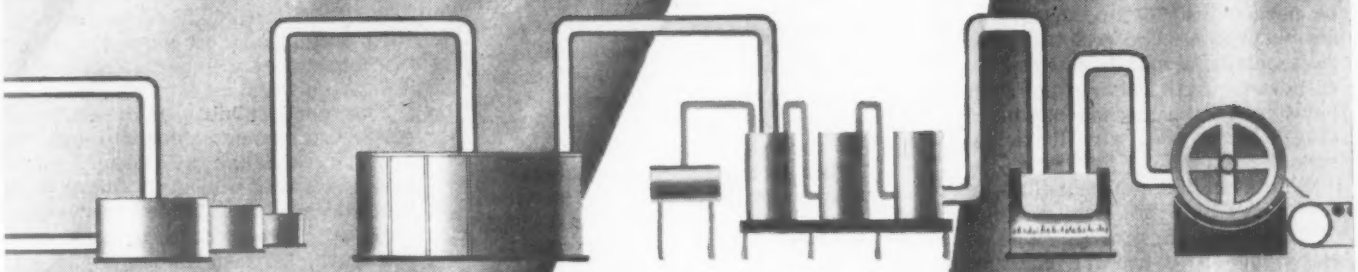

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is conventional in some respects but embodies numerous refinements both in equipment and operational details, incorporated to achieve a high degree of precision and accuracy.

The equipment employed in the cracking test is shown diagrammatically in Fig. 6, and consists of 3 main parts: a feed pump and controls; a cracking reaction system; and an automatic distillation apparatus.

Test equipment and procedure is detailed in the paper, and the selection, control and measurement of operating conditions is discussed. The method of arriving at activity and selectivity calibration curves is presented. With regard to other application of the test method the paper states:

"In addition to using the test method herein described to determine the activity ratings and selectivity factors of catalyst samples charged to or withdrawn from commercial or pilot-plant units, the method has been employed in connection with various research problems.

"These include the evaluation of new types of catalyst, determination of the effects of contaminants and accelerated deactivation treatments on various types of catalyst, determination of the effects of certain operating variables in catalytic cracking, and the determination of the relative ease of cracking and relative carbon- and gas-producing tendencies of various potential catalytic cracking feed stocks. The last-named application is becoming of increasing importance."

Standard Laboratory Method For the Determination of Cracking Catalyst Activity

By J. Alexander, Houdry Process Corp., Marcus Hook, Penna.

THE PAPER describes in detail the CAT "A" catalyst activity test which was developed in the Houdry laboratories in 1938, in response to the need for a quick, inexpensive method for evaluating and comparing different batches of experimental catalyst. By 1941 the test was being used not only in research and development work, but also for the control of catalyst manufacture and for following the aging characteristics and general behavior of catalysts in plant operation.

By 1944 variations of the method were used by a sufficient number of laboratories that its complete standardization was necessary. The standardization was accomplished cooperatively in the laboratory of the Houdry Process Corp. and that of the Socony-Vacuum Oil Co., at Paulsboro, N. J. At present the method is being used by more than 20 different laboratories.

The CAT "A" test is made in a small laboratory-scale cracking unit in which 200 ml. batches of catalyst are subjected to operating conditions chosen to simulate those of a commercial unit. Fig. 7 is a flow diagram of the apparatus.

The photograph on the cover of this issue is the CAT "A" catalyst activity test in operation in the Marcus Hook laboratories of Houdry Process Corp.

Apparatus and Procedure

The reaction chamber is made up of 2 connected sections of 25-mm. pyrex tubing, each fitted with a thermocouple well. The upper section is used as a vaporizer, and is filled with crushed quartz, a high heat-capacity material. The lower section, with a capacity of 200 to 220 ml. contains the catalyst to be tested.

The electrically-heated tubular furnace surrounding the reaction chamber consists of two sections of pipe, preferably separated by a thin layer of insulation. The heating elements are so arranged that the temperature differential from the top to the bottom of the catalyst bed does not exceed 10° F. under steady state conditions.

The test procedure through the cracking and regeneration cycle is described in the paper. The charging stock consists of a selected batch of light East Texas gas oil which has been carefully standardized. Test operating conditions are as follows: Preheater temperature, 830 to 850° F. Catalyst furnace temperature, 800° F. (± 5° F).

Charging rate, 1.5 volume of oil per volume of catalyst per hour, at 5 ml. ($\pm 0.3\%$) (milliliters at 60° F.) per 200 ml of catalyst per minute. Pressure, atmospheric.

Length of cracking period, 10 min.

The four numerical results necessary for a complete catalyst evaluation which can be determined or calculated by the test are:

1—Per cent by volume of motor gasoline.

2—Per cent by weight of catalyst deposit.

3—Gas gravity (referred to air).

4—Per cent by weight of gas.

For accurate catalyst evaluations it is the custom to make a sufficient number of cycles per catalyst to produce 3 gasoline yields within 1.5% of each other, 3 catalyst deposit yields within 0.3% and 3 gas yields within 1.0%. The averages of the acceptable results are taken as the final yields.

The reproducibility of the CAT "A" test has been evaluated through the periodic exchange of samples which are circulated to approximately 20 cooperating laboratories every three months. The average deviations as based on 22 samples tested in 9 to 19 laboratories, representing approximately 400 individual catalyst evaluations were as follows: $\pm$ 0.83% for the gasoline yield, $\pm$ 0.14% for the catalyst-deposit yield, $\pm$ 0.58% for the gas yield and $\pm$ 0.04% for the gas gravity.

In order to expand the utility of the present testing apparatus, two types of modifications have been investigated. One is the development of a standard heavy-stock activity test. Attempts have also been made to study the effects of changes in the cracking variables on certain catalysts by means of CAT "A" equipment.

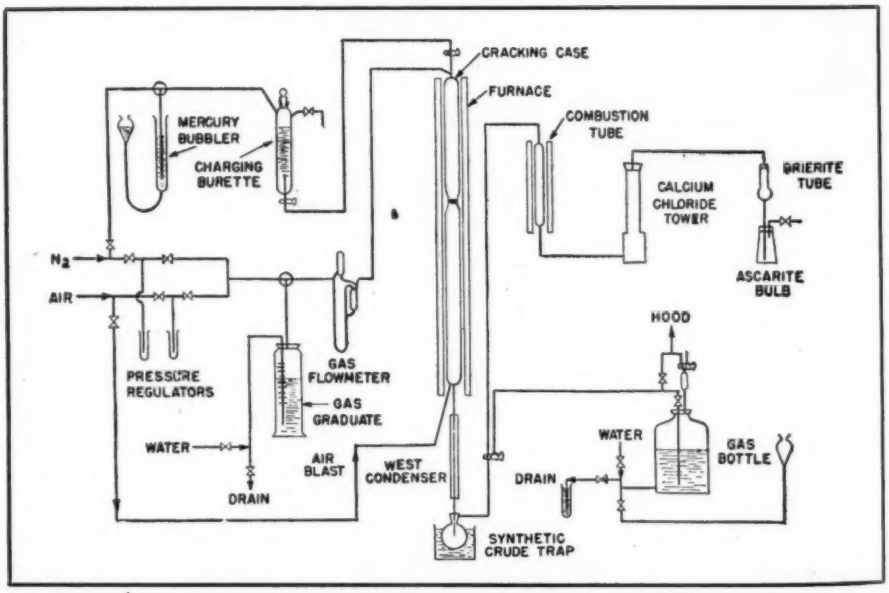


Fig. 7—Flow diagram of CAT "A" apparatus for determining catalyst activity

Small-Scale Laboratory Testing Of Cracking Catalysts

By G. F. Hornaday and D. B. Ardern, Houdry Process Corp., Marcus Hook, Penna.

A SMALL-SCALE fixed-bed catalytic cracking unit, with a catalyst capacity of 600 ml., was designed to give yield and quality data applicable to commercial installations. (This test unit is not to be confused with the so-called standard CAT "A" test method developed by Houdry which takes 200 ml. batches of catalyst).

The small-scale pilot plant consists of the oil, steam, and air preheater; catalyst case; charging pumps; condensers, receivers; and the necessary auxiliary equipment, such as temperature and pressure recorders, regulating valves, etc.

A sketch of the cracking case developed for this test is shown in Fig. 8 and the test apparatus is described. Principal steps in the operating cycle occur in the following order: 1—evacuation; 2—on stream; 3—pressure release; 4—oil purge; 5—regeneration.

Yield data from a cracking operation are first summarized in a material balance on a "condensed" basis, i.e., as produced from the unit. The condensed weight balance may be made over a single cycle or based on average data over a number of consecutive cycles. Equations are given for weight-balance calculations.

The catalyst deposit, in per cent by weight of charge stocks, is calculated from the results of the Orsat analyses on the regeneration fumes. The per cent by weight of wet gas is determined from the meter measurement, and the specific gravity is referred to air. Liquid condensates recovered in the hot and cold condensers and the vacuum trap are composited, and this condensed synthetic crude is weighed to complete the balance.

Liquid recovery, in per cent by volume of charge stock, is calculated from an API gravity measurement of the synthetic crude. The liquid product is analyzed for gasoline content by a "micro-still" distillation, which is essentially the same as for the CAT "A" test. In addition to the "condensed" material-balance-yield data, full "stabilized" yields are frequently obtained. For these data, complete light products yields through the C_4 or C_5 range are determined by obtaining mass-spectrometer analyses on samples of the wet gas and by Podbielniak distillations and analyses of cuts on samples of the liquid product.

As an example of a type of operation with the 600 ml. test unit, its use is described to define the aging characteristics of a sulfur stock on clay catalyst. A total of 90 cycles in the case produced contact of catalyst and oil equivalent to that expected in 18 days' operation with a TCC plant circulating 100 tons of catalyst per hour and having a total catalyst capacity of 480 tons.

This test unit is also used to explore the effect of certain variables in the cracking reaction from the standpoint of either the charging stock or the catalyst to be employed, since it can be operated over a much wider range of cracking variables than can be obtained in commercial units, while requiring only small amounts of charge stock and catalyst for the study.

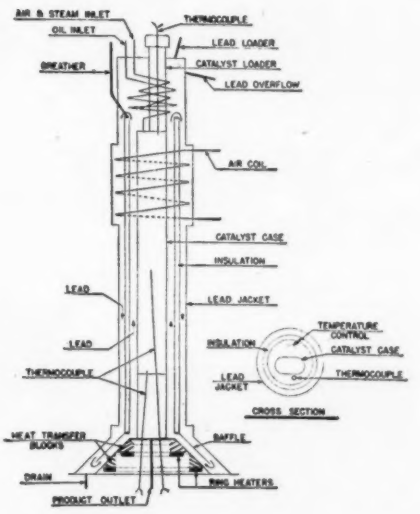


Fig. 8—Sketch of cracking case for Houdry small-scale pilot plant for testing cracking catalysts

Pilot-Plant Evaluation of Fluid Cracking Catalysts

By H. W. Grote and C. R. Olsen, Universal Oil Products Co., Chicago, Ill.

VARIOUS methods for routine testing of fluid cracking catalysts are in use in the UOP research and development laboratories. These tests are used to evaluate fresh catalyst as well as samples which have seen service in pilot-plant or commercial operations.

In addition to a wide range of laboratory tests, pilot-plant catalyst testing is carried on, the paper describing the pilot-plant testing as follows:

"At the present time, however, we feel that pilot-plant evaluation on a 3- to 4-bbl.-per-day scale is usually desirable on all catalysts that appear promising by the available laboratory test procedures. For pilot-plant evaluation work, we use units such as that shown diagrammatically in Fig. 9, the UOP fluid catalytic cracking pilot plant which has been described in some detail in past publications.

"These units can be operated at process and regeneration conditions which leave few uncertainties in comparing pilot and commercial scales of operation. In addition, specification gaseous and liquid product streams are produced which improve the reliability of product-distribution data.

"Catalyst evaluation tests in such equipment are run on a standard charging stock, and the program usually consists of a series of three or four once-through tests covering a wide conversion range plus a similar series of two or three recycle tests. In general, all these tests are made at constant reactor temperatures, and with the catalyst-to-oil

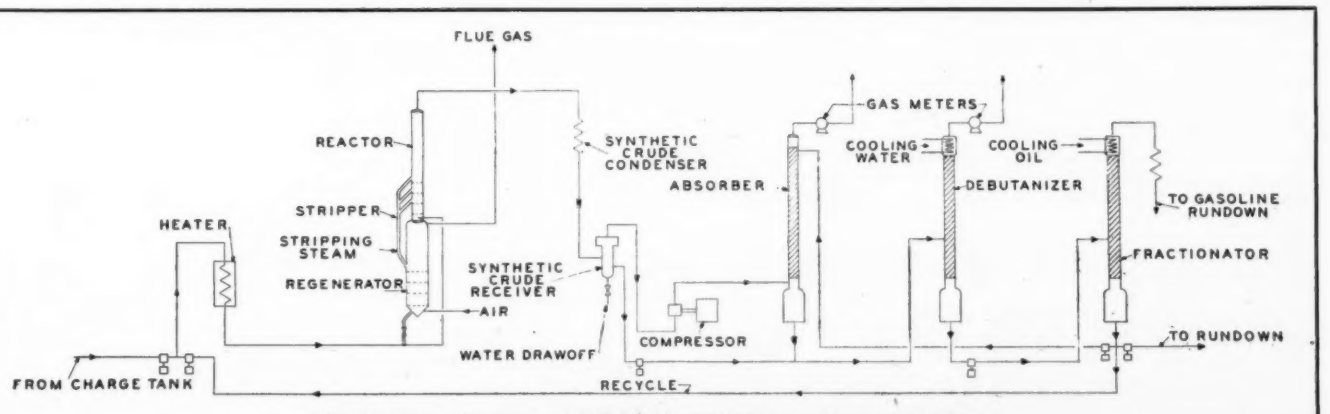
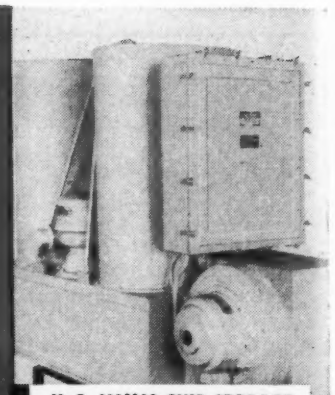


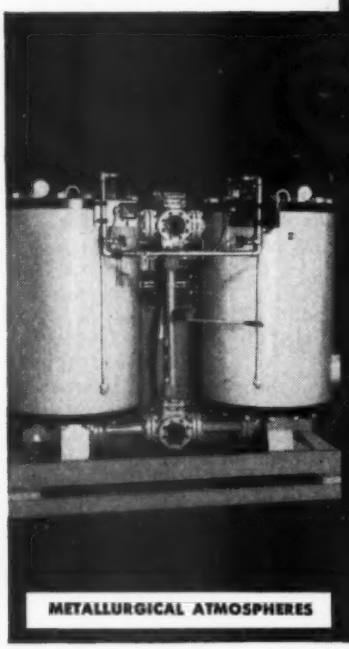
Fig. 9—Flow diagram of UOP fluid catalytic cracking pilot plant

MOISTURE - - - -

going, going, gone



U. S. NAVAL SHIP STORAGE



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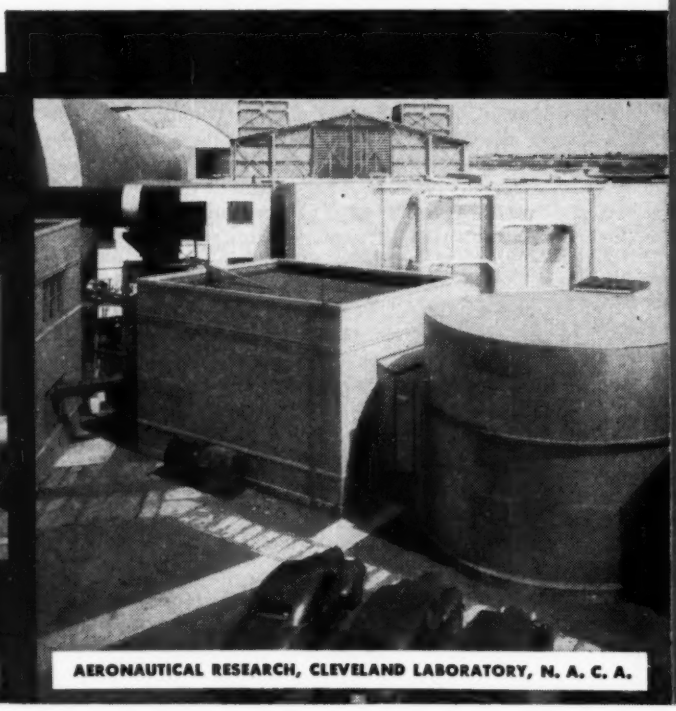
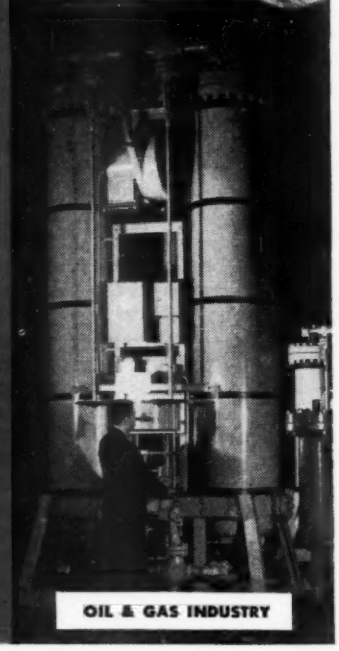


AERONAUTICAL RESEARCH, CLEVELAND LABORATORY, N. A. C. A.



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ratio being varied to maintain essentially a constant regenerator temperature. Reliability of such test results is satisfactory, with product recoveries averaging above 98.5% by weight of the feed stocks."

The paper presents product inspection data from pilot-plant operations which are not obtainable from standard laboratory evaluation tests. The standard single-cycle tests can be readily modified to determine gas compositions, it is pointed out, but these would be at very low conversions and, hence, of different composition than would be obtained in high conversion operations. Quantities of liquid products from laboratory tests also are not usually adequate for determination of gasoline and product gas-oil properties.

Data are presented pertaining to pilot-plant and commercial operations on the same charging stock while producing motor fuel. The catalyst and feed stock used in both scales of operation were the same. "The yield and product-quality data compared indicate satisfactory agreement between the two scales of operation", the paper states.

"The only appreciable difference in product distribution is in the catalyst-deposit yield, where the pilot plant shows a lower coke production —which is perhaps due to somewhat better stripping in the pilot-plant equipment. The commercial gasoline shows slightly lower octane ratings than does the corresponding pilot-plant product. These octane differences are probably due to difference in fractionation between the two scales of operation, as well as to the fact that, in the commercial operation, some slurry oil was returned to the reactor from the base of the fractionator. This resulted in a partial recycle operation and thus, an accompanying reduction in octane number."

Reprints of Catalyst Articles Available

In the past year PETROLEUM PROCESSING has published three special articles on catalyst properties, reporting some of the work done in the Riverside Laboratories of Universal Oil Products Co.

Copies of these reprints were distributed at the recent API meeting, and a few are still available for readers who were unable to get to the Chicago convention. Send your request to:

Reader's Service Department
PETROLEUM PROCESSING
1213 West Third St.
Cleveland 13, Ohio

Bibliography on Cracking Catalyst Testing

The combined literature references in the individual papers presented at the Symposium on Evaluation and Testing of Cracking Catalysts, of the API Refining Division, Nov. 10, at the annual API meeting in Chicago, make a useful bibliography on this relatively new research topic. The listing for all papers including literature references is given below for the convenience of readers:

"Plant Control Tests for Particle-Size Distribution of Fluid Cracking Catalysts," by K. D. Ashley and W. B. Innes, American Cyanamid Co.

(1) S. W. Martin, *Am. Soc. Testing Materials*, Symposium on Particle-Size Determinations in the Subsieve Range, Mar. (1941).
(2) J. M. Burgers, *Acad. Sci. Amsterdam 2nd Report on Viscosity and Plasticity* (1938).
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(5) F. M. Lea and R. W. Nurse, *J. Soc. Chem. Ind.*, 58, 277 (1939).
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(16) C. M. Chamot and C. W. Mason, *Chemical Microscopy*, Vol. I, 2nd edn., John Wiley and Sons, New York (1938).

"Modification in the Roller Analysis for the Determination of Particle-Size Distribution," by George L. Matheson, Standard Oil Development Co.

(1) P. S. Roller, *U. S. Bur. Mines Tech. Paper No.* 490, 46 (1931).
(2) P. S. Roller, *Trans. Am. Soc. Testing Materials*, 32, 607 (1932).
(3) P. S. Roller, *J. Am. Ceram. Soc.*, 20, 167 (1937).

"Accurate Particle-Size Determination of Fluid Catalyst by Coordination of Roller Analysis and Microscopic Examination," by John T. Wiley, Joe E. Deloney, and S. Walter Denton, The Texas Co.

(1) Paul S. Roller, "Separation and Size Distribution of Microscopic Particles—An Air Analyzer for Fine Powders," *U. S. Bur. Mines Tech. Paper*, 490 (1931).
(2) Herbert E. Schweyer and Lincoln T. Work, "Methods for Determining Particle-Size Distribution," *Symposium on New Methods for Particle-Size Distribution in the Sub-sieve Range, Amer. Soc. for Testing Materials* (1941).

"Prediction of the Activity of Cracking Catalysts from Heat of Wetting," by Ivor W. Mills, Sun Oil Co.

(1) J. Alexander and H. G. Shimp, "Laboratory Method for Determining the Activity of Cracking Catalysts—Standard Test Devised for Arriving at Yield of Motor Gasoline under Conditions Simulating Commercial Operation," *Natl. Petroleum News*, 36 (31) R-537 (1944).

"The Aromatic Adsorption Index as a Rapid Method for Approximating Catalyst Activity," by W. W. Scheumann and A. R. Rescorla, Cities Service Refining Corp.

(1) "Activity Test for Fluid Cracking Catalyst," *UOP Method M*-251-441, Universal Oil Products Co.
(2) J. Alexander and H. G. Shimp, "Laboratory Method for Evaluating Activity of Cracking Catalyst," *Natl. Petroleum News*, 36 (31), R-537 (1944).
(3) M. E. Conn and G. C. Connolly. "Testing of Cracking Catalyst," papers presented before Division of Chemistry, Am. Chem. Soc., Sept. (1946).
(4) "Physical, Chemical, and Catalytic Testing of Diakel Powdered Cracking Catalyst," *M. W. Kellogg Co., Petroleum Research Division*, June 7 (1943).
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"Determination of Activity and Selectivity of Cracking Catalysts," by Rodney V. Shankland and George E. Schmitkons, Standard Oil Co. (Indiana).

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(2) M. E. Conn and G. C. Connolly, "Testing of Cracking Catalysts," *Ind. Eng. Chem.*, 39, 1138-43 (1947).
(3) K. N. Anderson and M. J. Sterba, "Simplified Catalytic Cracking Unit Meets Requirements of Small Refiner," *Oil Gas J.*, 44 (33) 77 (1945).
(4) C. W. Siller, "Zinc-oxide Pigments—The Surface Area and Catalytic Activity," *J. Am. Chem. Soc.*, 65, 434 (1943).
(5) R. C. Hansford, "A Mechanism of Catalytic Cracking," *Ind. Eng. Chem.*, 39, 849-52 (1947).
(6) R. C. Davidson, "Cracking Sulfur Stocks with Natural Catalyst," *Petroleum Refiner*, 26, 663-72 (1947).
(7) E. W. Thiele, "Relation between Catalytic Activity and Size of Particle," *Ind. Eng. Chem.*, 31, 916-20 (1939).
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(9) G. M. Mood, H. H. Voge, and B. S. Greensfelder, "Catalytic Cracking of Pure Hydrocarbons," *Ind. Eng. Chem.*, 39, 1032-6 (1947) (and earlier papers).

"Bench-Scale Method for Determining Activity of Cracking Catalysts in Powdered Form," by Hubert McReynolds, The Texas Co.

(1) *ASTM Manual on Presentation of Data*, 3rd Printing, American Society for Testing Materials, Aug. (1940).
(2) M. E. Conn and G. C. Connolly, *Ind. Eng. Chem.*, 39, 1138-43 (1947).

"Design and Operation of a Bench-Scale Automatic Catalyst - Aging Unit," by T. Rice and F. E. Ivey, Jr., Gulf Research and Development Co.

(1) J. Alexander and H. G. Shimp, "Laboratory Method of Determining the Activity of Cracking Catalysts," *Natl. Petroleum News*, 36 (31) R-537 (1944).

"Pilot-Plant Evaluation of Fluid Cracking Catalysts," by H. W. Grote and C. R. Olsen, Universal Oil Products Co.

(1) P. H. Emmett and T. DeWitt, *Ind. Eng. Chem. Anal. Ed.*, 13, 28-33 (1941).
(2) G. M. Webb, *Petroleum Processing*, 2 (7) 497-502 (1947).
(3) Anon., *Oil Gas J.*, 45 (43) 82-3 (1947).

Patent Trends in Petroleum Refining

By PETER J. GAYLOR

Patent Attorney and Editor "The Technical Survey"

Developments disclosed in the patents reviewed below include:

Method for refining lubricating oils with sludging reagents and their separation

New spray type emulsifying agent for cleaning metals before coating

Separating Reagent Sludges

WHEN oils, particularly those in the lubricating range, are refined with sludge-forming reagents (such as sulfuric acid) at elevated temperatures, the sludge produced usually does not settle readily and is difficult to remove from the treated oil.

Atlantic Refining Co. has been issued U. S. Patent 2,127,589 covering a rather novel and useful method for accomplishing the separation. As shown in Fig. 1, the reagent and oil are first intimately mixed in agitator 1 and the mixture thus formed is allowed to fall in the form of a film over a heated drum 7.

This drum is heated to a temperature sufficient to coke or carbonize the sludge resulting from the reaction of the treating reagent with the oil, but insufficient to deleteriously affect the oil. The rate of supply of the mixture as well as the rate of rotation of the cylindrical surface is so controlled as to effect deposition of the coked sludge on the drum surface, while the treated oil 13 rolls off the drum in a direction opposite to that of rotation of the drum. The coke sludge adhering to the drum surface is continuously or periodically scraped off by scraper 11.

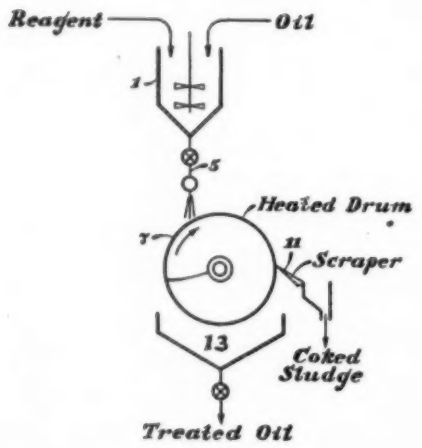


Fig. 1—Atlantic Refining Co.'s method for sludge removal from lubricating oils. U. S. 2,127,589

Usually, a drum temperature of about 400° F. to 600° F. is required when 66° Be' sulfuric acid is used for treating, although temperatures as low as 250° F. may be employed if the coking time is extended. With a reduced crude of 160 S.S.U. at 210° F. viscosity, and mixing in of 1% by volume of 66° Be' sulfuric acid, a yield of 88% decolorized, sludge-free oil may be obtained with an iron plate temperature of about 500° F.

Cleaning Metals for Coating

IT HAS been found, according to the American Chemical Paint Co., that cleaning compositions for preparing surfaces of iron, steel and aluminum for reception of paint, are not generally capable of removing completely greasy or oily matter. Such is the case, even if wetting agents are employed. Very often, the wetting agents cause the cleaning solution to spread over the top of the greasy surface so that even strong mechanical action is not able to make the solution penetrate the grease.

In U. S. Patent 2,396,776, a composition is described having a high oil and dirt removing efficiency. It may be applied by means of a spray without the troublesome foaming which is usually associated with emulsifying agents under such conditions.

The remarkable cleaning action is obtained by employing aqueous phosphoric acid solution which includes small quantities of water soluble, non-ionic emulsifying agents such as tertiary alkylarylpolyglycols.

An example of a concentrated cleaning composition to be diluted with water is as follows:

75% phosphoric acid	0.95 gal.
Emulsifying agent	0.16 lbs.
Water, to make	1.0 gal.

The amount of water used in diluting this formula depends on the amount of rust or oxide to be removed from the surface, as well as on the kind and amount of grease present.

For exceptionally greasy work, or when the cleaning solution is to be applied to the work by forceful spraying in a recirculating system, it is preferred to use a material in which is emulsified a water immiscible solvent such as petroleum naphtha, such as the composition given below:

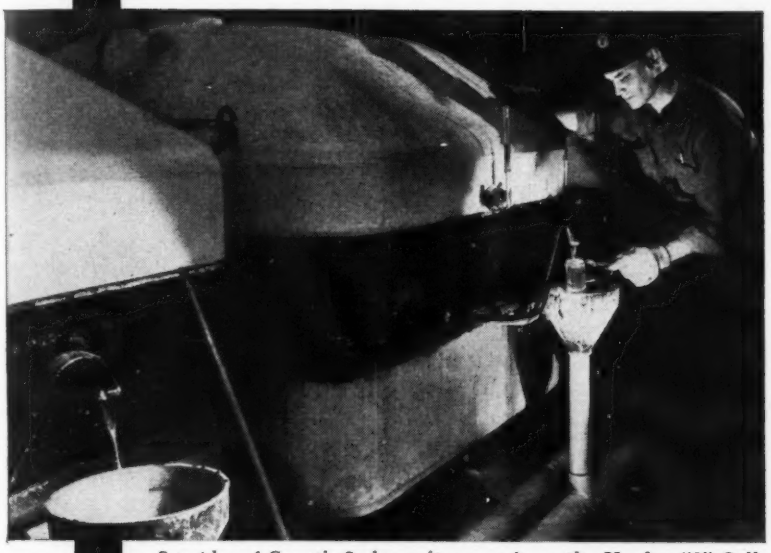
Phosphoric acid (75%)	0.100 gal.
Emulsifying agent	0.12 lb.
Stoddart solvent	0.625 gal.
Water, to make	1.00 gal.

This concentrate is violently stirred and passed through a homogenizer, or otherwise emulsified. With circulating sprays, it is diluted with 3-19 volumes of water.

Selected Patents of the Month

U.S. 2,422,670 & 4-5 (Universal Oil Products)—Selective demethylation of paraffinic hydrocarbons.
U.S. 2,422,671 (Universal Oil Products)—Lowering mol. wt. of non-aromatic hydrocarbons.
U.S. 2,422,672 (Universal Oil Products)—Triptane by selective demethylation.
U.S. 2,422,673 (Universal Oil Products)—Lowering mol. wt. of alkyl aromatics.
U.S. 2,422,692 (Universal Oil Products)—Conversion of normally gaseous olefin other than ethylene, to isobutane.
U.S. 2,422,769 (Monsanto)—E.p. lubricant..
U.S. 2,422,802 (Shell Dev.) — Dehydration of diacetone glycol to form a methyl pentadiene.
U.S. 2,422,859 (Phillips Petr.)—Nitriles of unsaturated acids.
U.S. 2,422,881 (Petrolite Corp.)—Compounded lubricating oil.
U.S. 2,422,884 (Shell Dev.)—Isomerizing olefins with boric oxide on alumina catalyst.
U.S. 2,422,991 (Anglo Iranian)—Isohydropolymers from olefins.
U.S. 2,423,013 (Socony)—Contacting gases with particle form solid materials.
U.S. 2,423,029 (Houdry Proc.)—Diolefins.
U.S. 2,428,067 (Atlantic Refining) — Ammonia and soap for breaking nitrobenzene oil emulsions.
U.S. 2,428,119 (Texaco Dev.) — Oxygenated compounds by treating olefins with sulfuric acid.
U.S. 2,428,123 (Cities Service) — Soda base grease.
U.S. 2,428,235 (Shell Dev.)—Aromatic hydroxy ethers.
U.S. 2,428,417 (Kellogg)—Alkylation
U.S. 2,428,455 (Phillips Petr.)—Olefin isomerization.
U.S. 2,428,506 (Shell Dev.)—Alkylation
U.S. 2,428,516 (Phillips Petr.) — Isobutylene separation.
U.S. 2,428,590 (Shell Dev.)—Allyl acetate by reacting propylene with selenium dioxide dissolved in acetic acid.
U.S. 2,428,623 (Standard Oil Development)—Regenerating spent caustic.
U.S. 2,428,668 (Standard Oil Development) — Sulfuric acid olefin absorption.
U.S. 2,428,686 (Standard Oil Development)—Treating feed stock with aryl sulfonic acid.
U.S. 2,428,692 (Standard Oil Development)—Isobutane and isopentane by destructive hydrogenation of oils.
U.S. 2,428,695 (Pure Oil) — Hydrocarbon extraction.

Three Hooker Chemicals That Help Make Thousands



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Construction Economies Are Found In Improved "Fluid" Cracking Unit

SEVERAL ECONOMIC features are claimed for an improved design for a catalytic cracking unit of the fluid type recently patented.

Among them are: 1) reduction in overall height by making reactor and regenerator concentric chambers, 2) internal standpipes, eliminating most of the large, costly expansion joints, minimizing heat losses, and reducing stresses and strains because of the usual temperature changes encountered in conventional external piping.

The unit is shown diagrammatically in Fig. 1. Flow is indicated by the arrows, and is of the concurrent type. Hot oil charge enters at 71, picking up regenerated catalyst from standpipe 21 at valve 26, and passing up through the internal reaction zone 10. Separation is provided by a series of cyclones 12, product vapors passing on to the recovery system through outlet 15.

Spent catalyst from the conversion zone overflows into standpipe 14 and passes into regeneration chamber 11 through valve 16. Regeneration effluent gas is separated from the fines in cyclones 18 and leaves via line 28. The catalyst then completes the cycle by passage down standpipe 21.

Conventional stripping and aerating steam, and air lines are indicated as is the temperature-controlling steam coil in the regeneration zone. The efficient use of heat transfer from the exothermic burning zone to the endothermic conversion zone is self-evident from the diagram.

When using powdered catalysts, reactor vapor velocities of 0.4 to 4.0 ft./sec. are preferred. Catalyst/oil ratio should be about 8 or 10 to 1 when cracking reduced crude. Catalyst residence time may range from a few seconds to an hour or more, and the vapor residence time usually about 10 to 30 seconds.

U. S. 2,428,872, issued October 14, to Robert C. Gunness, assignor to Standard Oil Co. (Indiana), Chicago, Ill.

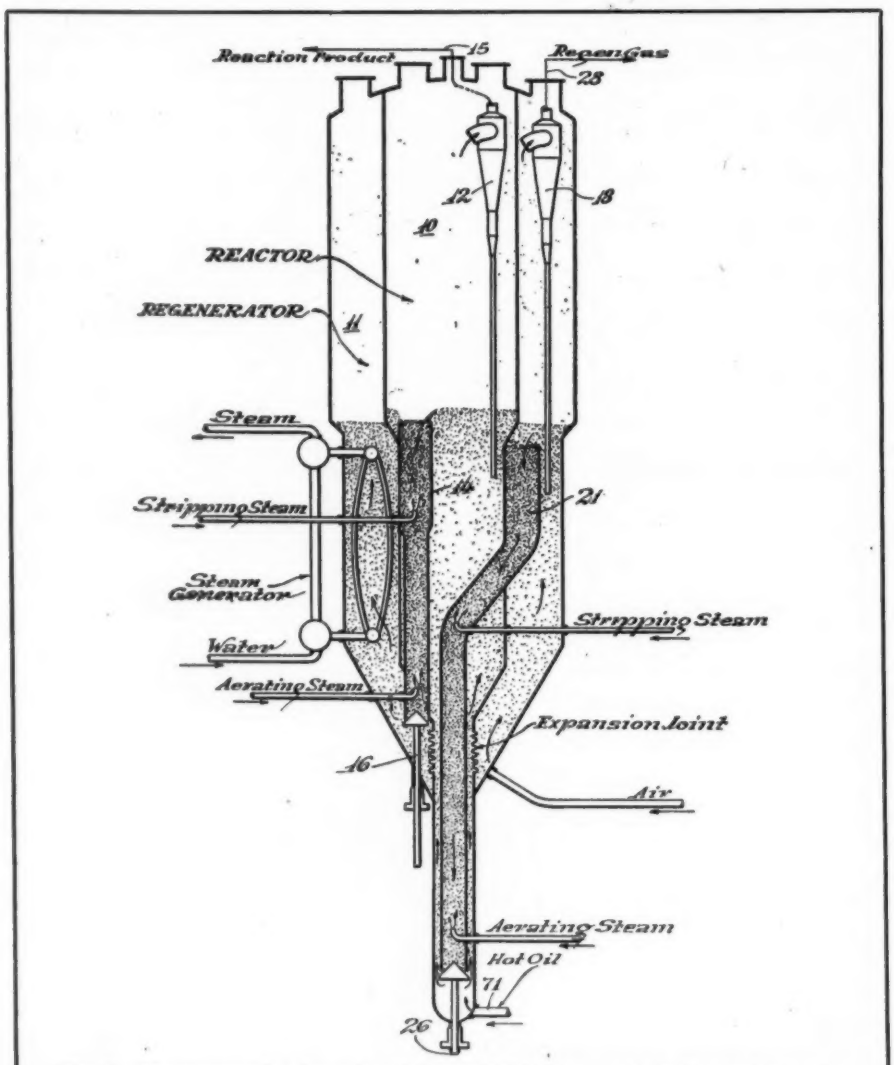


Fig. 1—Diagrammatic view of an improved fluid type catalytic cracking unit (U. S. 2,428,872)

Notched Trays Stop Channeling In Packed Towers or Reactors

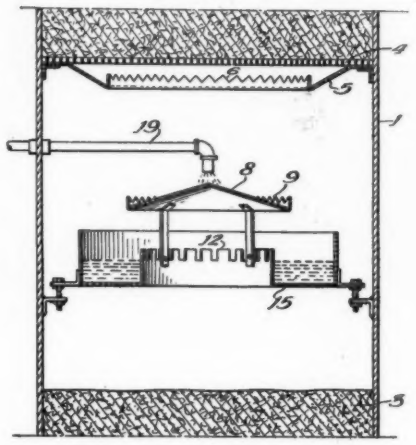


Fig. 3—Solid streams of downflowing liquid are broken up by passage over notched trays 5, 8, and 15 (U. S. 2,428,922)

UNDESIRABLE CHANNELING by down flowing liquid in packed towers or in contact beds in a reaction chamber can be eliminated by installing distributing trays of proper design at intervals throughout the height of the vessel. In addition, the distributing devices improve the efficiency of feed from incoming intermediate streams.

Chief function of the baffles or trays as shown in Fig. 3 is to break up any solid streams of downcoming liquid into tiny streams, allowing, at the same time, sufficient cross-sectional area for flow of upward-moving vapors. Designed primarily for use in caustic regenerators of units for removing mercaptans from gasoline, the device will serve equally well for any packed chamber, it is stated.

Liquid in cylindrical column 1 first strikes ring-shaped baffle 5 and flows over notched weirs 6, thence to ring-shaped tray 15 and again over notched weirs 12 to lower bed of packing 3. Sidestream enters through line 19 and correspondingly is broken up by flowing over inverted saucer 8 with notches 9.

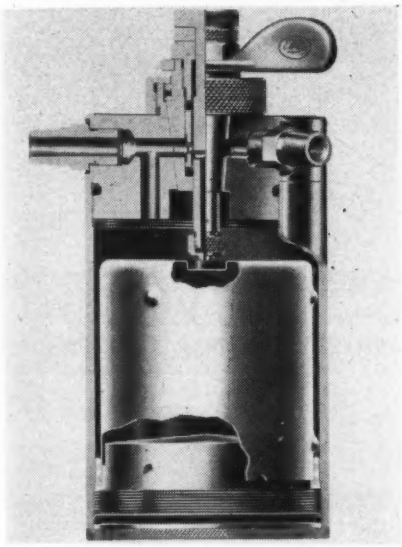
U. S. 2,428,922, issued October 14, to Allen M. Shoresman, assignor to Universal Oil Products Co., Chicago.

What's New!

Edited by
WILLIAM C. UHL
Equipment Editor

EQUIPMENT . . . MATERIALS . . . PROCESSES . . . LITERATURE

1—Pressure Regulator



Physical principle of the Cartesian diver is used in the design of a new automatic pressure regulator, the Greiner Industrial Cartesian Manostat Model 5, developed from a glass manostat for laboratory use (see "What New!", October, pge. 789, Item 12). The instrument uses three interchangeable orifices, will maintain constant pressure or vacuum to within 0.1%. All metal, it weighs 6½ lbs., measures 7½ in. overall by 3¼ in. diameter. Emil Greiner Co.

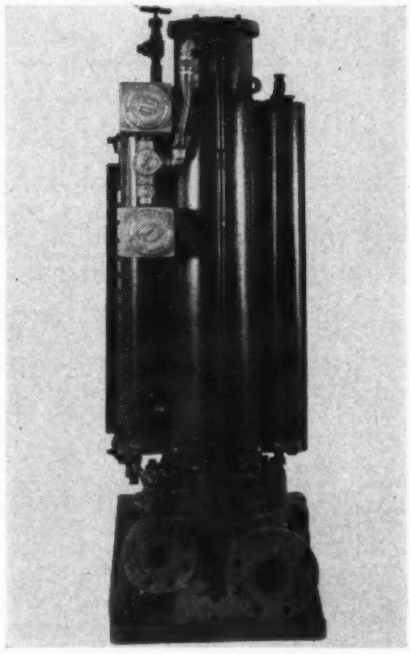
2—Thread Compound

Prevention of freezing and galling of metal surfaces at temperatures as high as 1800° F. is claimed for the new Fel-Pro "Hi-Temp" Thread Compound C-5. The material was developed specifically for high pressure and temperature applications such as furnace tube header studs and plugs, according to the manufacturer, where it will reduce expensive stud breakage. Felt Products Mfg. Co.

For More Information

Use one of the attached business reply cards, which requires no postage, to request additional details or literature on any items reviewed in "What's New!" Just circle the numbers corresponding to the numbers on the items you're interested in, fill in the bottom of the card, and drop it in the mail.

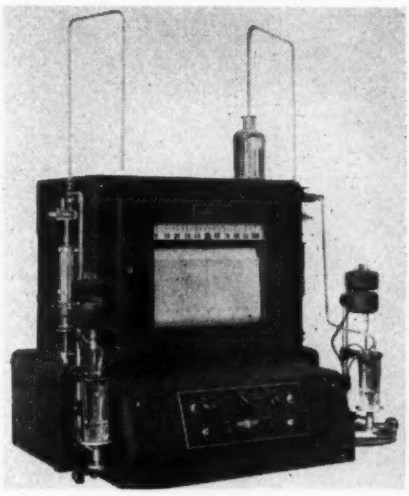
3—Stuffingboxless Pump



Elimination of leakage with its attendant fire-hazard and high maintenance cost is said to be a feature of the B&J Stuffingboxless pump, an electric motor driven centrifugal pump. It is especially applicable to installations outdoors and for unattended automatic operation. (See "What's New!", November, pge. 877, Item 17). Byron-Jackson Co., Pump Division.

4—Recording Titrator

The new "Precision"-Dow Dual Recordomatic Titrator is said to be the first of its type designed for plotting automatically titration curves. The instrument is recommended for both routine control and research work, for acidity determinations on petroleum products, mercaptan determinations, the study of indicators, etc. Operator is required only to prepare solutions and load the feed unit; while one sample is being titrated, a second may be prepared. A modified Brown Electronik potentiometer is used to record and control analyses. Five meter scale and potentiometer circuits give a total range of minus 1.5 to plus 1.5 volts, and a full pH range of 0 to 14. The instrument is line-operated except for No. 6 dry cell in the potentiometer circuit. Power consumption is 200 watts and the Titrator is suitable for continuous duty. Precision Scientific Co.



5—Nylon Filter Cloth

Nylon filter cloth is now available, and can be had in twill, chain, and plain weaves in numerous porosities and in widths from 26 to 72 in. It can also be supplied in the form of a made-up filter element with all sewing done by Nylon thread. Nylon filter cloths are said to have high abrasion resistance, and heat resistance (melting point about 482° F.), and are unaffected by micro-organisms. They have a smooth surface which facilitates cake discharge, and are inert to many acids, caustics, and organic compounds. Filter Media Corp.

More "What's New!" Items, Page 963

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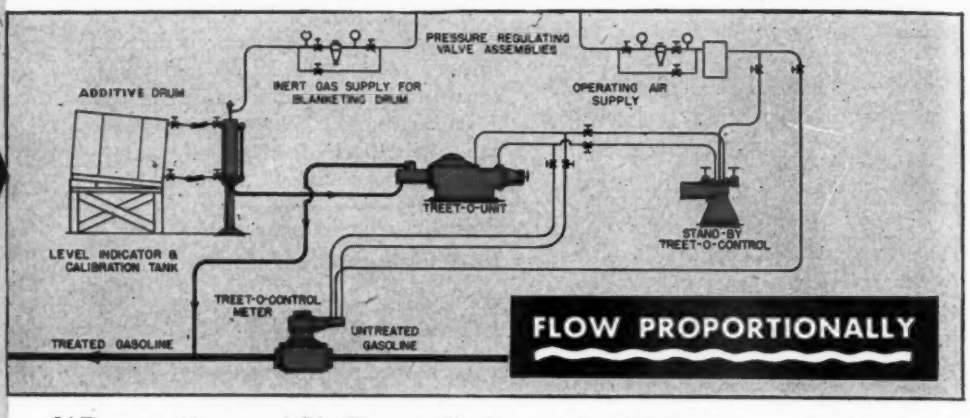
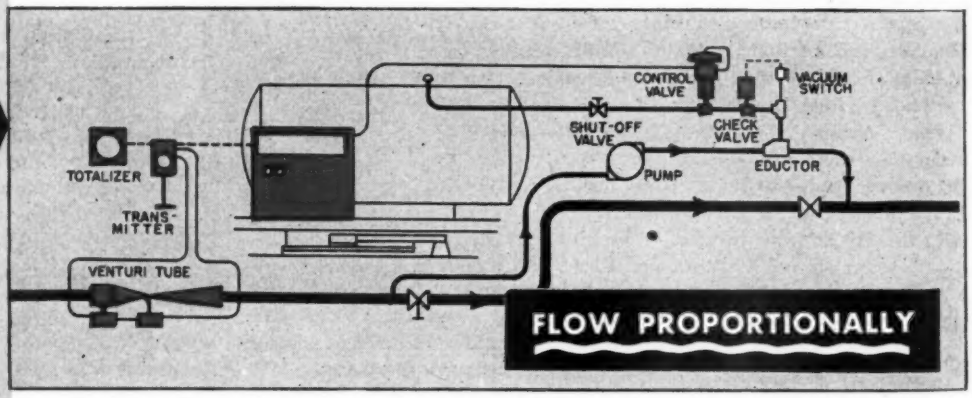


%Proportioneers% Treet-O-Control offers an economical and accurate method of applying to petroleum products additives such as treating agents, inhibitors, lubricants, dyes, etc. Equipment is available for flow rates from 5 to 5,000 G.P.M. and injections of from .0005 to 15 G.P.M.

BY WEIGHT



%Proportioneers% Lead Blender delivers an accurately proportioned, homogeneous mixture of gasoline and Tetraethyl Lead with minimum cost and wastage. Mixing of fluid and gasoline takes place continuously in the line. No batch blending, with attendant equipment and rehandling expense, is required.

%Proportioneers% methods permit wide flow variation with sustained accuracy, and additives are applied automatically. As specialists in the field . . . with over 25,000 installations of proportioning equipment . . . we offer engineering advice and the latest equipment for continuous processing and blending. Ask for Bulletin REF.

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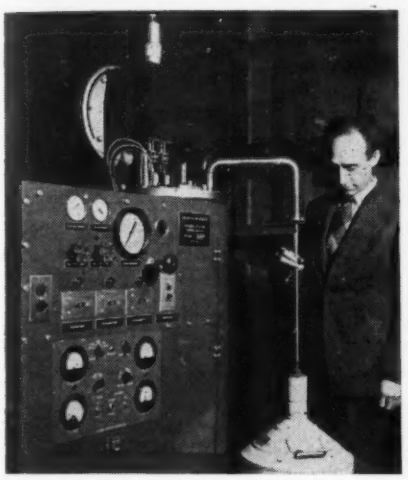
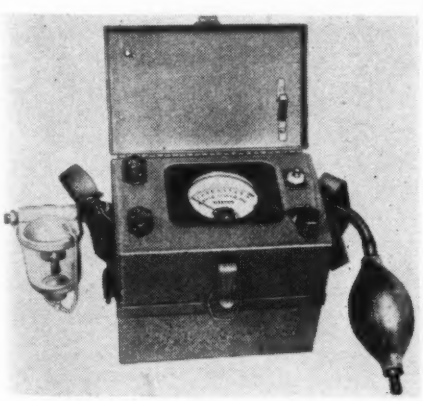
6—Low Temperature Apparatus



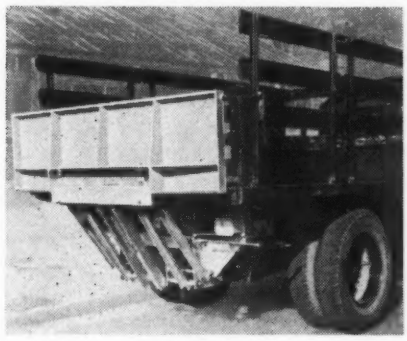
Research work at temperatures close to absolute zero is now possible with the Collins Helium Cryostat, which liquefies helium, and can maintain any temperature down to 2° Kelvin, or minus 456° F. Invented by Dr. S. C. Collins of Massachusetts Institute of Technology, the Cryostat has been developed into a design which can be operated by laboratory technicians without attention by scientific personnel. Complete unit weighs about 2800 lbs. and occupies less than 50 sq. ft. of floor space. Helium is the only refrigerant used. The first Cryostat went into use early in 1947. An oil company is among the three users of the seven units now in operation. Arthur D. Little, Inc.

7—Flammable Gas Indicator

The Vapotester Model 6 is a new two-range instrument which will detect gases in their toxic range in addition to indicating the lower explosive limit of combustible concentrations of flammable gases or vapors. Operating principle of the Vapotester is the use of a Wheatstone bridge circuit to compare differences in resistance of two filaments, one completely sealed within a chamber, the second housed in an open chamber through which the gas to be analyzed is passed. The two ranges are provided by a three-position switch. One position is for the combustible range, one is for checking the voltage, and the third to increase the sensitivity of the first scale ten times. Davis Emergency Equipment Co.



8—Truck Loader



The Day hydraulic tail gate loader for trucks is furnished in a complete package and is said to take only a few minutes to install. The hydraulic pump is driven from the truck's standard power take-off and is controlled by a convenient lever, or the pump can be operated by hand. The combination tail gate-lift platform operates over the full distance from ground to truck floor level, handling loads up to 1200 lbs., and remaining level at all points in its travel arc. Unit fits most 1½ ton trucks. The Day Co.

9—Potentiometer

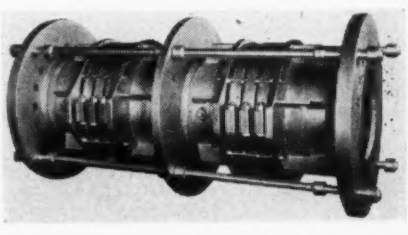
Accuracy and sensitivity are assured in a new series of temperature-calibrated portable potentiometers, with four models offering improved sensitivity in the low temperature ranges, according to the manufacturer. Instrument includes special structural features which maintain the thermocouple reference-junction and the automatic reference-junction compensator at substantially equal temperatures, irrespective of changes in the ambient temperature. Units are well suited for use in petroleum and chemical processing where small temperature differentials are of interest. A total of 10 standard models are available covering a wide temperature range, for use with copper-constantan, iron-constantan, or chromel-alumel thermocouples. Special ranges can be supplied; calibration may be had in either Centigrade or Fahrenheit scales. Rubicon Co.

10—Vinyl-lined Pipe

Developed for handling strong chemicals is a new plastic-lined pipe using Vinyl resins. Pipe is lined by first extruding the plastic in the form of a tube with a wall-thickness of 0.050 to 0.060 in. Tube is then placed inside steel pipe and bonded to it. Amercoat Division of American Pipe & Construction Co.

11—Expansion Joint

Particularly suited for catalyst lines because it has no sharp crevices in which catalyst can lodge, the Zallea Universal Expansion Joint is said to have applications in reactor risers and standpipes, regenerator standpipes, and stripper and flue gas lines. It consists of two corrugated elements linked by a section of pipe. The elements can be either of non-equalizing type for pressures up to 30 psi., or self-equalizing for higher pressures. Zallea Bros. & Johnson.



More "What's New!" Items, Page 964

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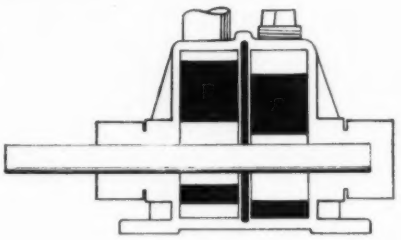
12—Gearless Pump



A double-impeller design is said to be the feature of a new line of small "ECO" gearless pumps adapted to handling acids, solvents, oils, and the like. The double-impeller provides a strong, smooth flow against pressure, according to the manufacturer. Pump bodies are available in stainless steel, monel, or bronze. Pump sizes are ¼ and ½ in., capacities from one to 12 gpm. Pumps can be run and will deliver at speeds from 200 to 3500 rpm., and can be used against pressures up to 50 psi. Eco Engineering Co.

Trade Literature

13—pH Recorder

Bailey pH Recorder, Bulletin No. 234, gives operating and design description of the new Bailey pH Recorder. Bailey Meter Co.

14—Arc-Welding

Arc Welding is being Handicapped, a reprint of an article in the *Scientific American* by James F. Lincoln, presenting the arguments for and against the use of arc-welding vs. riveting methods for joints. Lincoln Electric Co.

15—Flow Measurement

Flowrator, Catalog Section 10-D, explains operating principle of Flowrator instruments for liquid and gas flow measurement and control. Illustrations of typical applications. Fischer & Porter Co.

16—High Vacuum

High Vacuum Apparatus, Bulletin 10, a 48-page catalog including engineering data helpful in the design of high vacuum systems, covering such topics as pumping speed, connections, merit factor, gages and traps. Central Scientific Co.

17—Pneumatic Transmitter

Model 42 Pneumatic Transmitter, Advance Bulletin No. 409, provides a description of the new transmitter in both eccentric and concentric types. The Foxboro Co.

18—Clad Steels

A Visit to Lukens, a 28-page booklet telling in pictures and words an interesting story of how stainless steel and various other alloys are made into clad steels, and including the manufacture of unusual shapes from heavy plate by flame-cutting and welding processes. Lukens Steel Co.

19—Knock Testing

Publications No. 15-46 *and* 15-47 describe the operation and specifications of Knockometers for automotive and aviation fuel testing on single cylinder laboratory engines, covering Model K-1 (aviation) and K-3 (automotive). Sperry Gyroscope Co., Inc.

20—Demineralized Water

Filt-R-Stil Demineralizers, 28 pages of descriptive data on equipment designed to produce demineralized water, a low-cost chemical equivalent of distilled water. Units covered range in capacity from 8 to 1000 gpm. American Cyanamid Co.

21—Manhole Covers

Catalog 11 is a 20-page booklet covering forged boiler and tank accessories, manhole covers and yokes, handhole covers and yokes, welding street ells and water column ells, including information about curving handhole covers. Steel Improvement & Forge Co.

22—Gas Separator

Campbell Screenless Mist-D-Fier, a four-page bulletin explaining the principle of operation of an improved gas separator which will remove crude oil from natural gas entering an absorption plant, or oil mist and water from gas leaving the plant, as examples of a number of uses listed. J. A. Campbell Co.

For Your Convenience

Business reply cards are included in this issue of PETROLEUM PROCESSING to assist you in obtaining more information on any items reviewed in "What's New!" You'll find them facing page 961. Just circle the numbers corresponding to the numbers you're interested in, fill in the bottom of the card, and drop it in the mail. No postage required.

New Solutizer Unit Has Unique Features For Disulfide Removal and Regeneration

THE Tannin Solutizer unit recently placed in operation at Cities Service Oil Co.'s Ponca City, Okla. refinery has two unique points to distinguish it from units installed elsewhere. The problem of removing the heavier disulfides from the treating solution has been conquered (this plant is one of the first to treat a relatively heavy gasoline fraction), and it represents the first use in a refinery of graphite air diffusers in the regeneration step.

The "Solutizer" process has been described in the literature by La-Croix[1, 2] and others, including the addition of tannin as an additional promoter.[3] Briefly, the process calls for the use of a strong caustic solution containing isobutyric acid (as sodium or potassium isobutyrate) as a promoter to increase solubility of mercaptans. Tannin also is included as an organic oxidation promoter for the regeneration phase of the continuous process to convert soluble mercaptans to insoluble disulfides.

Feed for the new unit consists of 1200 b/d of a light pressure gasoline (thermally cracked) with an initial boiling point of 100° F. and an end-point of 300° F. Mercaptan sulfur content ranges from 0.0162 to 0.0216%. The plant's thermal gasoline is split three ways, the C_4 cut being caustic washed for hydrogen sulfide removal, and the 300-400° F. cut being Doctor-treated, since its relatively small mercaptan content is of too high a molecular weight for satisfactory removal by the Solutizer method.

Solutizer Treating

"Solutizer" feed enters the system at atmospheric temperature following a light wash with caustic to remove hydrogen sulfide not included in the C_4 cut previously mentioned, to avoid weakening the Solutizer solution. The extractor is arranged to counterflow raw gasoline and treating solution in intimate contact, followed by a coalescer section at the top where treated gasoline is broken out for settling and storage. Gasoline leaving the settler contains about 0.0030% mercaptan sulfur.

The system contains 170 bbls. of "Solutizer" solution. Each barrel represents 84.3 lbs. of lye, 92.6 lbs. of isobutyric acid and 0.25 wt./% of solution as tannic acid. The resulting solution is 6 Normal as caustic, 3 N. as sodium isobutyrate.

A simplified flowsheet of the process is shown in Fig. 1. Fat Solutizer solution emerges from the bottom of the extractor and enters the regenerator at 125-130° F. after picking up heat by exchange with steam. This column is not packed and the solution blown with plant air for regeneration. Regenerated solution is bottoms and the spent air and some disulfides vent to a stack after passing a mist extractor (side-to-side baffles).

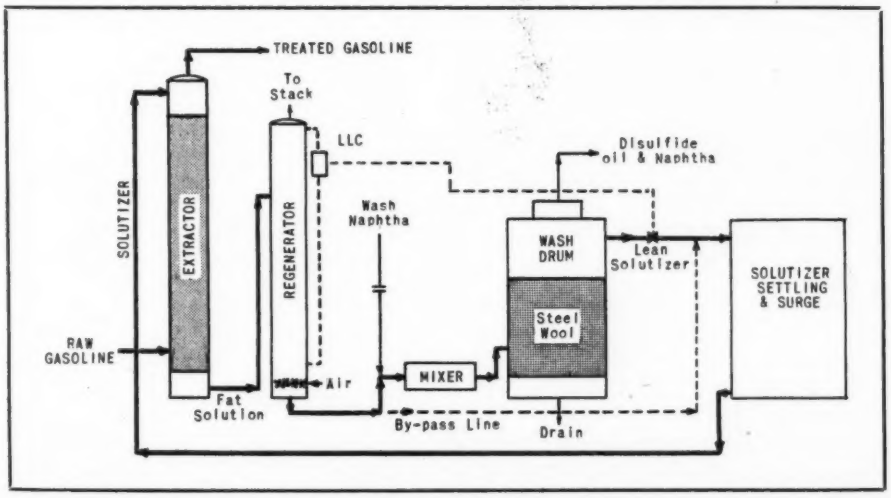


Fig. 1—Simplified flowsheet for Tannin Solutizer sweetening unit at Ponca City refinery of Cities Service Oil Co.

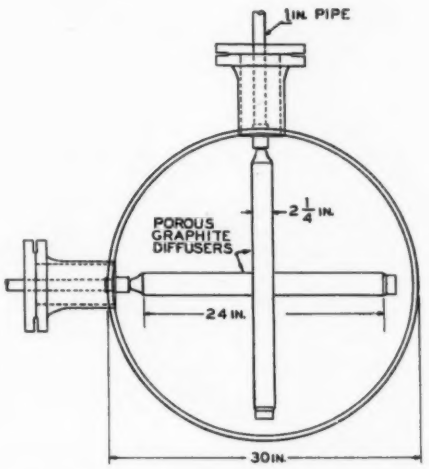


Fig. 2—Section of regenerator tower just above air diffusers, showing dimensions and arrangement

The regenerator is low carbon steel throughout, no monel or other alloy equipment being required. This is made possible through the use of the organic oxidizing agent tannin, thus permitting regeneration at 120-150° F. temperatures instead of the customary 200° F. and higher. The sulfur compounds present are not corrosive to steel at this low temperature.

The use of porous graphite to diffuse regeneration air marks a "first-time" application for the oil industry, it is believed, although it has been used in sewage disposal plants for several years. Temperatures are below that at which oxidation of carbon occurs and the tube is inert to caustic or sulfur attack. The highly porous tubes produce much smaller air bubbles with proportionately greater opportunity for intimate contact than possible with previous diffusion nozzles. A sectional view of the regenerator appears as Fig. 2, showing dimensions and arrangements of the diffusers. Plant air at about 8 to 12 psi is used, with flow rate of 14-15 cu. ft. per minute.

Sulfur Oil Removal

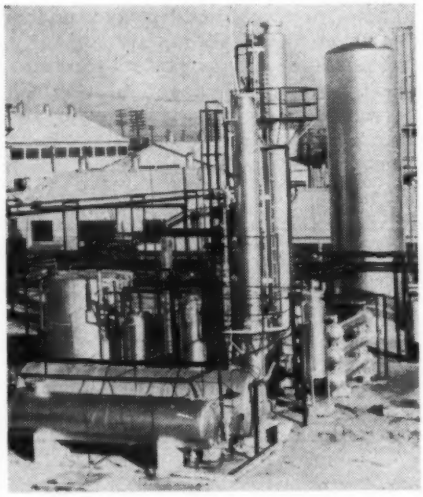
After the unit was installed and shake-down operation started, it was quickly noted that, due to the heavy mercaptans present, disulfides were not settling out of the regenerated "Solutizer" solution. Consequently,

ostensibly treated gasoline actually was emerging with a considerable disulfide content and reduced lead susceptibility. The reason was the relatively high specific gravity of the disulfides and, while they could be removed by diluting the solution with water — "springing" — a considerable cost-factor is involved in reconcentrating the solution, for which the plant is not equipped.

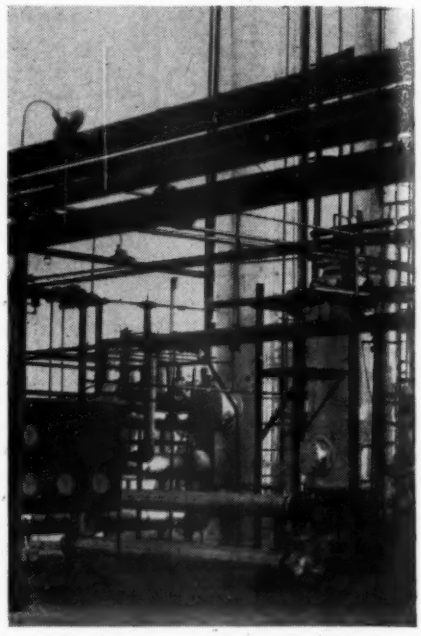
Several expedients were tried. Washing the regenerated solution with a small quantity of fresh heavy naphtha (250° F. IBP. and 350 endpoint) removed this material satisfactorily. At first a drum was used, containing 6 bbls. of naphtha through which the spent solution was forced. A new batch of naphtha was required every 24 hours.

This batch washer has since been replaced with a Duriron mixer to handle the two streams. This well-mixed stream then is charged continuously to a 3 ft. by 7 ft. drum filled with steel wool, which promotes separation of the two fluids. The wash naphtha is taken overhead while the now truly lean solution ends in a combination settling and surge tank for return to process.

The wash naphtha from the drum is charged directly to the thermal reforming unit. At first it was feared that increased corrosion would be evident in the reformer, as well as an increased sulfur content in the reformed gasoline. To date no corrosion has been noted and apparently the disulfides are completely converted to hydrogen sulfide, as there has been no noticeable increase in mercaptan content of reformer gasoline to date.



View of compact Solutizer unit. Vessels visible, left to right, are raw gasoline surge tank, wash naphtha surge tank, regenerator and extractor. Small vessel immediately in front of extractor is the disulfide wash drum; Solutizer storage is hidden by the columns. Large vertical vessel in rear and two horizontal vessels in foreground are parts of other nearby units



Another view of the Solutizer unit, showing simple control panel, extractor and regenerator

Low-Cost Treating Saves Lead

A worthwhile saving in tetraethyl lead requirements in these days of short supplies has been effected by Solutizer treating at Ponca City. For every gallon of Solutized gasoline 0.46 cc less lead are required to bring the octane rating to 74 (ASTM Motor method) than was the case with Doctor treating. In practice the saving is greater since treating is carried out in a closed system, whereby most of the vapor losses formerly experienced in batch agitators are eliminated. Calculations indicate this saves at least 0.5% of gasoline throughput.

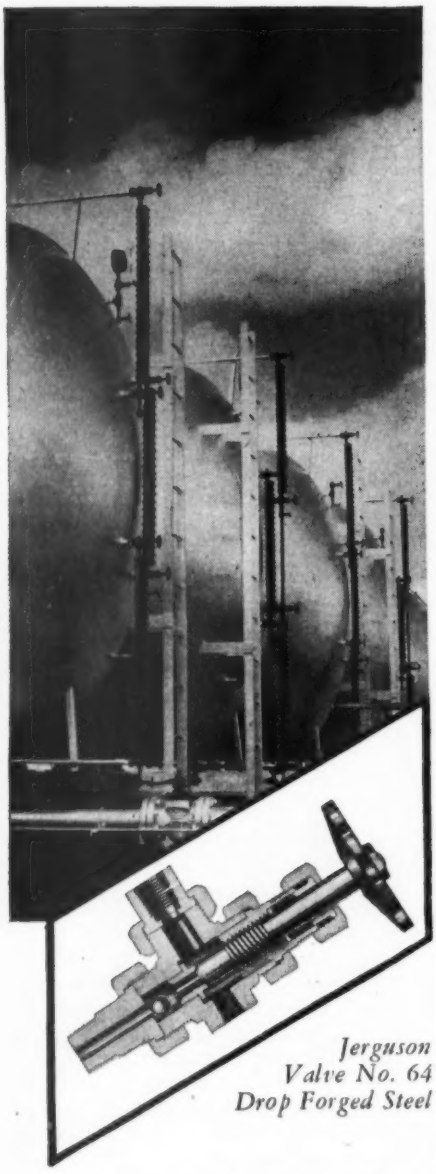
Solutizer operating costs at Ponca City have been slightly higher than for the Doctor treating formerly used. Part of this is from generally higher labor and materials cost, and the more expensive initial treating chemical charge. These greater costs are amply justified by tetraethyl lead savings and reduction in product loss during treating, according to company engineers.

References

(1.) LaCroix, H. N., "Solutizers Improve Treating of Four Types of Gasoline", *National Petroleum News*, 33, No. 10, p. R-72-80 (1941).

(2.) ———, "Improved Refining of Sour Crudes by New Tannin Solutizer Process," *NPN Technical Section*, 36, No. 9, p. R-170-171 (1944).

(3.) Anon., "Tomorrow in Petroleum Technology—Tannin Solutizer Process Continues to Show Possibilities," PETROLEUM PROCESSING, 1, No. 1, p. 7-8 (1946).



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PAPER

Sour Crudes—Equipment Protection*

By E. Q. CAMP

Humble Oil and Refining Co., Baytown, Texas

This paper presents a discussion of some of the major corrosion problems experienced in refining sour crudes. The corrosion that occurs in the storage, transportation, and distillation of sour crudes and sour distillates is predominantly low-temperature corrosion (below about 500° F.). The life of tanks in sour-crude service is increased by gunite coatings, coatings of paints and plastics, caustic washing, heavier steel construction, use of aluminum and galvanized iron roofs, and wetting roofs with crude oil. Of these, gunite coatings are probably the most satisfactory. Neutralizers, gunite coatings, and corrosion-resistant alloys are used to control corrosion of distillation equipment.

High-temperature corrosion (above about 500° F.) is predominant in cracking service. The protective measures employed in this type of corrosion include extensive use of corrosion-resistant alloys, gunite coatings, and linings of alloys, as well as the use of neutralizers in charge and product.

CORROSION is one of the major problems confronting the designer and operator of refinery equipment. It has been estimated that the losses due to corrosion in the refinery industry alone are equivalent to approximately 1 cent per gallon of gasoline produced.(1) These enormous losses occur as a result of the large number of corrosion problems experienced in refining. These problems include essentially all corrosion common to other industries and, in addition, a number of corrosion problems peculiar to the refining industry. The latter are primarily those which arise as a result of the presence of corrosive sulfur compounds and of chlorides of magnesium and calcium in the crude oils. The major offenders in this respect are the high sulfur crudes, frequently referred to as sour crudes, which include West Texas and Panhandle.

The corrosion experienced in processing sour crudes has been discussed in detail in a number of papers presented on this subject during the past several years.(2 to 22) Although the present paper is limited primarily to the experiences with corrosion in one refinery, and to corrosion mitigation in processing sour crudes, it is emphasized that these experiences closely parallel those in other refineries in which sour crudes are processed; as a result, many of the problems discussed and the solutions presented in this paper have already appeared in the literature on this subject.

CLASSIFICATION OF CORROSION

(This section of Mr. Camp's paper is omitted here. In it he classifies corrosion as either low-temperature —below about 500° F., or high-temperature—above about 500° F., and then discusses in more detail the chemicals in a sour crude which are responsible for both types of corrosion and the extent of their effect.

(The low-temperature corrosion experienced in processing sour crudes, he points out, is due primarily to hydrogen sulfide, hydrochloric acid, water, oxygen, and carbon dioxide.

(Corrosion of the high-temperature type is due primarily to sulfur compounds present in the crude oil. The presence of inorganic salts, particularly chlorides of calcium and magnesium, may have a marked effect upon the rate of corrosion experienced. Of the various sulfur compounds present in sour crudes, Mr. Camp continues, it is generally agreed that free sulfur, hydrogen sulfide, and mercaptans are the most corrosive under high-temperature conditions.

(The balance of Mr. Camp's paper, presented below, is devoted to a discussion of some of the major corrosion problems encountered in Humble's Baytown refinery in storing, transporting, distilling, and cracking sour crudes and distillates, and of the methods employed in mitigating these problems.)

TANKS

The corrosion of steel tanks in sour-crude and sour-distillate services is a problem of considerable significance in the operation of a refinery.(24) The corrosion is due to the action of hydrogen sulfide, oxygen, and water. The roof and two top rings of the shell usually suffer the most corrosion. The corrosion of the bottoms of the tanks presents the next most important problem. The corrosion of the shells of the tank, other than the two top rings, presents the least problem. The roofs of tanks in sour-crude service have a life varying from about 5.3 to 11.6 years, with 8.7 years being the average life. The roofs of tanks in sour-distillate service have a life from 3.8 to 14.3 years, with an average life of 7.9 years.

Means of Increasing Tank Life

Heavier steel construction: It is indicated that one of the most effective low-cost methods of increasing the life of a tank is to increase the thickness of the steel plates. As an example, it is common practice to use 3/16-in. plates for tank roofs. Based upon replacing the roofs when the plates reach a minimum thickness of 1/16-in., increasing the original thickness of the plates by 1/16-in. offers an increase of 50% in the life of the roof for a 33% additional cost of material for the roof, or approximately a 20% additional overall cost for material and labor; because, due to the longer life expected from the thicker plates, the yearly cost for installation and dismantling would, of course, be considerably reduced. As a result of the longer relative life of the rafters and other structural supports as compared with that of a roof of normal thickness, the use of thicker roof plates would more nearly equalize the replacements of these units of equipment. The use of 5/16-in. roof material could probably be justified for use on rundown tanks and tanks of intermediate size in sour service where the previous roof lasted less than about 6 years; whereas 1/4-in. material would probably be more practical on larger tanks, where

*(Presented before the 27th Annual Meeting of the American Petroleum Institute, Chicago, Nov. 11, 1947)

increased thickness of supporting members would be a factor.

Roofs of aluminum and galvanized steel: Twenty-seven aluminum roofs have been installed on rundown tanks in sour-distillate service at Baytown. The first of these roofs was installed in March, 1928. All of the others were installed between February, 1931, and February, 1936. No corrosion has been experienced on these roofs proper, other than the formation of a thin white coating, probably of aluminum oxide. Some galvanic corrosion has been experienced at the point where the roofs are riveted to the angle irons connecting the roofs and shells of the tanks. The same type of corrosion has been experienced where the roofs contact other steel supporting members.

In December, 1930, two rundown tanks were equipped with galvanized roofs. These roofs are still in good condition. The only evidence of corrosion is a white powdery material formed on the surface of the galvanized coatings.

The present practice is to install steel roofs on rundown tanks, rather than aluminum or galvanized roofs; because the average life of steel roofs is approximately 8.2 years, and an aluminum or galvanized roof, either of which costs approximately 2 to 3 times as much, would have to give at least 16 years of uninterrupted service to pay out the added investment. Further, steel roofs can be protected at a relatively low cost.

Caustic washing: Caustic washing of distillates from sour crudes has effected an increase of 5.4 years in the average life of tank roofs in rundown and intermediate storage service. Maximum use is made of this method of increasing the life of tank roofs, because it is eventually necessary to remove hydrogen sulfide from sour distillates.

Flat roofs in crude service: It has been observed that the roof and top rings of tanks in sour-crude service corrode more rapidly than do the shells. This difference in corrosion rate is attributed to some extent to the protective film of oil left on the shells when the tanks are emptied after having been filled. In some cases flat roofs are installed on tanks in crude service with provisions for filling the tanks and wetting the underside of the roofs once every 30 days, thereby maintaining a protective film of oil on the roof plates. This procedure has the advantage of reducing the vapor space of the tanks to a minimum, which is reported to be effective in reducing the corrosion of the roof plates.

Because of the apparent advantage to be derived from wetting the underside of the roof plates with crude oil, spray systems have been installed in some tanks whereby crude oil may be sprayed at regular intervals on the roof and the top shell plates, thereby maintaining a protective film of oil on these portions of the tanks.

It is indicated in some cases that the practice of wetting the roofs and shells of tanks with crude oil has effected an increase in the life of these portions of the tanks, but it is not conclusive at present that this type of protection is economically feasible for general application. This is due primarily to: 1, the high cost of the equipment required to spray or wet the roofs and other portions of tanks; 2, the difficulties experienced in dislodging the water film from horizontal surfaces; and, 3, interruptions in pumping schedules in order to wet the roofs at intervals of 3 to 15 days, the frequency required to obtain any appreciable protection.

Protective coatings: A wide variety of nonmetallic materials has been tested by application to sand-blasted steel panels and exposure to sour vapors in crude and rundown tanks. Direct application of those materials showing the best protection were made to the roofs of tanks in sour service. None of these materials has given satisfactory protection for as long as two years. It is concluded from this work that none of the protective coatings manufactured at present is sufficiently resistant to justify its application to the roofs of tanks in sour-crude and sour-distillate services.

Gunite linings: Tank bottoms in sour-distillate service have been protected with concrete for about 18 years at the Baytown refinery. The first applications of concrete were made to the tank bottoms themselves, but later applications included a gunite lining extending about 18 in. up the shells of the tanks. Because of the good results obtained with coatings of this type, gunite coatings were applied to the entire shells, roofs, and structural members of several tanks. The results obtained were satisfactory, and it is now common practice to gunite the inside surfaces of essentially all tanks in rundown and intermediate sour-distillate service.

The guniting of a tank is accomplished in three separate operations. These consist of the preparation of the surface, installation of the reinforcement, and application of the gunite. The final cleaning of a tank in preparation for guniting consists of sand-blasting to bare metal. It is common practice to sand-blast new tanks to remove the mill scale.

The preparation of a tank for gun-

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iting, the installation of the reinforcing, and the application of the gunite coating have been discussed in detail in the literature.(25)

The cost of applying a gunite coating is in the order of 50 to 60% of the cost of a new tank. The application of gunite coatings to a tank is justified on the basis that they at least double the life of the tank. Normally, it is expected that gunite linings will more than double the life of a tank. The maximum life of such coatings in sour-crude and some distillate services has not been established.

LINES

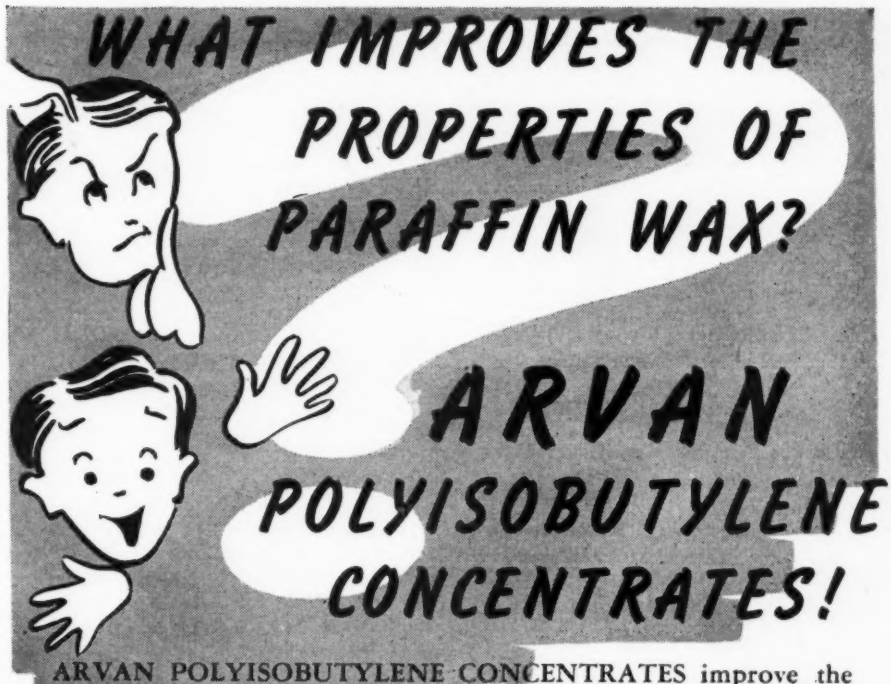
It is not general practice to protect lines in sour-crude and distillate services, although it is recognized that they are subject to all the corrosive conditions that usually affect tanks in similar services, except that possibly less air is present in the lines. However, in some isolated cases ammonia or caustic solution may be injected into distillate lines to reduce corrosion. In those cases the corrosion is usually due to an inorganic acid, such as hydrochloric acid, rather than to compounds of sulfur.

DISTILLATION EQUIPMENT

The data presented in this paper on the corrosion encountered in fractionating sour crude are based on the experience obtained in reducing West Texas crude to about 40% bottoms in a pipe still. The corrosion experienced in this particular operation parallels that experienced in other distillation operations in which sour crude constitutes the charge. It is believed, therefore, that a discussion of the corrosion experienced in this particular operation will suffice for the corrosion experienced in the distillation of sour crude in general.

A simplified flow diagram of the pipe still used is shown in Fig. 1. Briefly, the charge of West Texas crude flows through a steam preheater, through naphtha vapor-to-crude and bottoms-to-crude exchangers, and is then flashed in the flash and reservoir section of the primary tower; the stabilized crude flows into the furnace. The stabilized crude is heated to a temperature of about 575° F., and flows from the furnace into the primary tower, where light naphtha is distilled overhead. The bottoms from the primary tower enter the secondary tower, where the remainder of the upper 60% of the crude is removed as overhead and side streams. The bottoms from the secondary tower are pumped through the bottoms-to-crude exchangers and then into a reduced-crude cracking unit.

In the early operation of this pipe still, rapid corrosion was experienced in the furnace tubes and overhead



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ARVAN 612-6	133°-135° AMP Fully refined	151-194 cs when diluted to 50% with wax used in mfg.	53%	Low
ARVAN 612-7	(White Petroleum Jelly)	— —	— —	Medium

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Product	Melting Point	Viscosity*	% Vistanex†	Mol. Wt. Vistanex†
ARVAN 613-1	170°-175° AMP Bareco B Square Amber	120-155 cps	20%	High
ARVAN 613-2	155°-160° AMP Socony 2310	55-85 cps	20%	High

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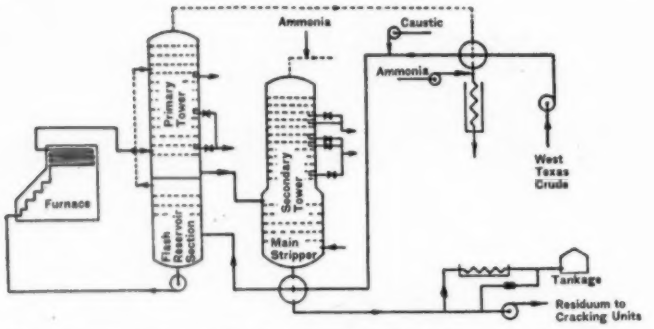

Fig. 1—Flow diagram of pipe still used in reducing West Texas sour crude at Humble's Baytown refinery

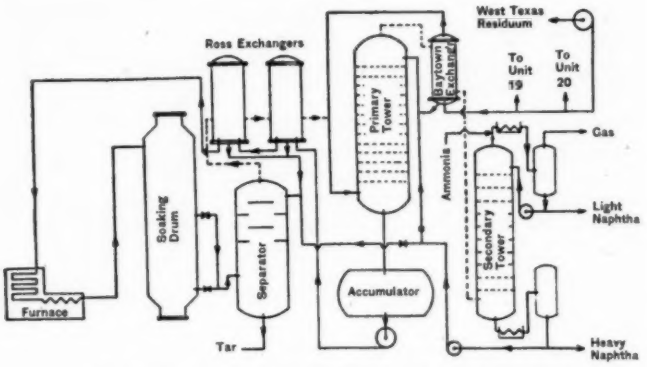

Fig. 2—Flow diagram of residuum cracking unit

lines and condensers. Further details of this corrosion and the methods employed in its mitigation are presented in the following discussion.

Furnace Tubes

All the original tubes in the furnace of this pipe still were of low-carbon steel. Rapid corrosion of these tubes was experienced, necessitating replacements after 4 to 6 months. The first step in reducing this corrosion consisted of introducing caustic with the crude charge. It was found that caustic solution equivalent to 6 lb. of sodium hydroxide per 1000 bbl. of crude oil could be injected with the charge without affecting the operation of the pipe still or of the residuum cracking units. This quantity of caustic reduced the corrosion of the furnace tubes, but replacements of tubes near the coil outlet was necessary after 6 to 8 months. Larger amounts of caustic progressively reduced furnace-tube corrosion at the pipe still, but limited operations at the residuum cracking units because of the rise in tube-metal temperature of the cracking-coil furnace tubes. As a result of this, caustic solution equivalent to 6 lb. per 1000 bbl. of charge was injected with the crude charge, and 20 steel tubes at the coil outlet were replaced with 4- to 6% - chromium - 0.5% - molybdenum tubes. This combination of injecting caustic with the crude charge and using 4- to 6%-chromium tubes near the furnace outlet has controlled satfactorily the corrosion of the furnace tubes.

The fact that the injection of caustic with the charge to the pipe still reduced the corrosion of the furnace tubes indicates that the corrosion was due in part to chlorides in the crude charge, particularly hydrochloric acid formed by the hydrolysis of calcium and magnesium chlorides at elevated temperature.

Condensers

In the initial operation of this pipe still, rapid corrosion of the vapor lines and condensers was experienced. This corrosion was due to hydrochloric acid formed as a result of the hydrolysis of calcium and magnesium chlorides present in the crude charge. The injection of caustic with the crude charge effected a marked reduction in the amount of hydrochloric acid evolved, but did not eliminate it. The vapor lines are of low-carbon steel, the naphtha-vapor-to-crude exchanger contain admiralty - metal tubes in a steel shell, and the final condensers and coolers are cast iron of the coil-in-box type.

Ammonia is injected into the vapor lines upstream from the coil-in-box condensers to control the corrosion of this equipment. Ammonia injection and its control have been discussed on numerous occasions. In this particular case, the pH of the water condensed with the naphtha is maintained at 6.5 to 7.0. Water is injected upstream from the ammonia-injection point to prevent the deposition of ammonium salts in the vapor lines and condensers. Higher pH's are avoided to conserve ammonia. If higher pH's were maintained considerable quantities of the ammonia would be consumed in neutralizing hydrogen sulfide. This is of no particular significance in corrosion control under conditions of this type.

Frequently, ammonia is injected upstream from exchangers that are equipped with copper-bearing alloy tubes. It is normally assumed that copper-bearing alloys in condensers on sour-distillate streams would be corroded rapidly by ammonia when this neutralizer is injected upstream from such condensers, particularly if the pH of the water condensed with the sour distillate is maintained above the neutral point. It has been found in practice, however, that this is not the case. Several observations have been cited in an attempt to explain this apparent anomaly, but no satisfactory explanation has appeared in the literature. The results of some recent work on this phase of the problem indicate that certain sulfur compounds, such as hydrogen sulfide, mercaptans, carbon disulfide, etc., are very effective inhibitors for controlling the corrosion of copper-bearing alloys in atmospheres containing ammonia and oxygen. The results presented below show the extent to

Compound Added	% Added	Corrosion Rate, In./Yr.	Reduction of Corrosion Rate, %
None	...	0.5895	...
Ammonium hydrosulfide	0.025	0.0368	93.8
Ammonium hydrosulfide	0.05	0.0187	96.8
Ammonium hydrosulfide	0.10	0.0320	94.5
Ammonium hydrosulfide	0.30	0.0096	98.4
Butyl mercaptan	0.25	0.3930	33.4
Butyl mercaptan	0.50	0.0678	88.5
Butyl mercaptan	2.0	0.0442	92.6
Butyl mercaptan	2.5	0.0315	94.5

which sulfur compounds reduce the corrosion of admiralty metal in a 10%-by-weight solution of ammonium hydroxide at room temperature for 48 hours.

In another case, admiralty metal under stress failed as a result of stress-corrosion cracking in an atmosphere containing ammonia, moisture, and oxygen after 3 hours of exposure. This test was repeated with 3.0% by weight of butyl mercaptan added to the ammonium-hydroxide solution over which the stressed admiralty metal was suspended. After 194 hours of exposure, there was no evidence of failure as a result of stress-corrosion cracking. Equally good results were obtained with hydrogen sulfide and carbon disulfide.

CRACKING UNITS

In selecting material for presentation and discussion on the corrosion encountered and on the methods employed in its mitigation in cracking reduced sour crudes and sour distillates, the corrosion experienced in the operation of three separate types of cracking processes has been chosen. These processes include thermal cracking of reduced West Texas crude, cracking of sour distillate in a fluid catalyst cracking unit, and vapor-phase reforming of a light naphtha. The discussion of the corrosion experienced in these cracking processes is limited to some of the major corrosion problems involved, because it would be beyond the scope of this paper to discuss in detail all the corrosion experienced. It is believed that the corrosion discussed in con-

nection with these three cracking processes is representative of the major corrosion problems encountered in cracking in general.

Thermal Cracking Unit

Reduced West Texas crude from a pipe-still operation is cracked in a partial-yield operation on a thermal cracking unit. Fig. 2 shows a flow diagram of this type of cracking operation. The residuum charge flows through a heat exchanger and is discharged into the bottom of the primary distillation tower, where it combines with recycle stock, and then flows into the hot fuel accumulator. The total feed from the accumulator is pumped at a pressure of about 1200 psi through heat exchangers, and then into the furnace coil. The feed flows from the furnace coil at a temperature of about 900° F. and a pressure of about 700 psi downflow through a soaking drum. The feed then flows through a pressure-reducing valve and is discharged into a separator, where tar is removed at the bottom and the light products pass overhead through heat exchangers, and then into the primary distillation tower. Recycle stock flows from the bottom of the primary distillation tower into the total feed accumulator. The overhead from the primary tower passes through heat exchangers and then into the secondary distillation tower, where it is separated into light and heavy naphtha.

Furnace tubes: The first 72 tubes in the convection section of the furnace are low-carbon steel, the next 30 tubes are 4- to 6%-chromium steel, and the last 16 tubes in the convection section and the 39 tubes in the radiant section are 18-8 alloy. Originally all tubes in this furnace were of low-carbon steel, but those in the radiant section and the last 16 tubes in the convection section were replaced with an 18-8 alloy because of the rapid corrosion experienced. It was later found necessary to replace the next 30 tubes in the convection section with 4- to 6%-chromium steel tubes. The life of steel tubes in the radiant section of the furnace was as low as 30 days, and the life of the last 16 tubes in the convection section was in the order of 3 months.

In general, the performance of 18-8 alloy tubes in this type of cracking operation has been satisfactory. Many of the original tubes are still in service after more than 100,000 hours of operations. On the basis of certain examinations, it has been postulated that the deterioration and, possibly, the failure of the 18-8 alloy tubes is due to structural changes at the grain boundaries. These changes are believed to be progressive in nature, and are dependent upon time, temperature, and stress. It is further postulated that these structural changes are due to the decomposition of the austenite, which results in the precipitation and growth of highly alloyed ferrite. When these precipitated areas have reached a certain size, cracking occurs under certain conditions with respect to time, temperature, and stress. Once cracking has occurred, the tube is permanently brittle, but at any time prior to the cracking the tube is either ductile or can have ductility restored by proper heat treatment.

Hot-oil lines: In the initial operation of this plant, rather severe corrosion was experienced in all lines through which oil was pumped at a temperature above about 450 to 500° F. In order to eliminate this type of corrosion, essentially all of the lines downstream from the furnace have been replaced with 18-8 alloy lines.

Soaking drum: The soaking drum is of low-carbon steel. The temperature conditions are such that rapid corrosion would occur if the drum were not protected. The drum is protected against corrosion by the application of a reinforced layer of gunite about 3 in. in thickness. This type of protection is very effective in re-

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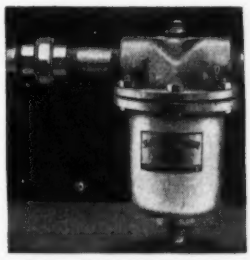
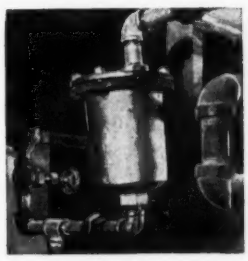


Super-Silvertop showing one nipple and one union needed.



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ducing corrosion of the drum, but the coating is frequently damaged in the process of removing coke from the drum. This necessitates frequent repairing and replacing of the gunite lining. This type of protection is so effective that these drums are in satisfactory operating condition after almost 20 years of service in cracking reduced West Texas crude.

Separator: The separator was originally of all-steel construction. Rapid corrosion was experienced in this piece of equipment, and it was necessary to apply protection to avoid failure of the separator. The protection employed consists of guniting the shell and installing an 18-8 alloy lining in manhole openings, etc. The plates in the separator are of steel, but are being replaced with 4- to 6%-chromium steel as they fail. All supports are of 18-8 alloy.

Bubble towers: The bubble towers are of low-carbon steel, and are equipped with steel plates and cast-iron bell caps, except for the three bottom plates in the primary bubble tower. These plates are of 11- to 13%-chromium steel, with bell caps of the same material. The shell of the primary tower is protected with a gunite lining.

Accumulator: The accumulator is of all-steel construction, and is protected with a gunite lining.

Preheaters: The exchanger is the primary preheat circuit, and is of all-steel construction. Corrosion is not a problem in this preheater. The exchangers in the secondary preheat circuit are equipped with steel shells and 18-8 alloy tubes. The steel shell is protected by metallizing with aluminum. This protection is satisfactory and, as a result, maintenance costs are low.

Neutralizers: It was found that the injection of caustic, 6 lb. per 1000 bbl. of crude, into the crude charge to the pipe still resulted in an appreciable reduction in the corrosion of the thermal cracking unit in which the residuum from the pipe still was processed. Larger quantities of caustic could be tolerated in the pipe still, but any increase in the amount of caustic injected, i.e., above 6 lb. per 1000 bbl. of crude, materially affected the service factor on the thermal cracking unit as a result of the rapid rise of metal temperatures in the furnace tubes. It was found, however, that an increase in the caustic injected into the charge to the pipe still effected a corresponding decrease in the corrosion of the cracking equipment. An experimental study of lime injection into the cracking unit was made to determine whether the corrosion of cracking equipment (occurring while 6 lb. of caustic per 1000 bbl of crude was being injected into the charge to the pipe still) could be reduced further by injecting lime ahead of the cracking-unit furnace. It was found that, when 250 lb. of lime per 1000 bbl. of residuum was injected, the life of the steel tubes in the convection section of the cracking-unit furnace was increased by about 40%, and the overall loss of iron from the unit was decreased by 20 to 30%. The injection of lime was never put into practice, because the increase in the sediment content of the fuel from the cracking operation was more than could be tolerated.

Ammonia is injected into the overhead vapor line from the secondary bubble tower to neutralize inorganic and organic acid and, thus, reduce the corrosion of the overhead vapor line and condensers as well as other equipment downstream from the cracking unit.

Fluid Catalyst Cracking Unit

Corrosion, as such, is not a problem of any particular significance in the operation of Fluid catalyst cracking units. Some difficulty is experienced as a result of erosion of certain portions of equipment, but serious corrosion has not been experienced except in one case. This corrosion occurred in the 18-8 alloy vapor lines leading to and from the reactor on an upflow unit, and was intergranular in nature. The vapor lines in which this stress-corrosion cracking occurred were lined at points of turbulence with carbon-steel wear plates to absorb erosion caused by the catalyst. The intergranular cracks that occurred in these vapor lines were located behind the carbon-steel wear plates.

In the investigation of this corrosion problem, it developed that there was a small annular space between the wear plates and the 18-8 alloy vapor lines. Catalyst and hydrocarbon vapors circulated at low velocity in this space when the unit was on stream. During this time, the carbon-steel wear plates were corroded rather rapidly by hydrogen sulfide at the operating temperature of about 950° F. As the velocities in the annular space between the wear plates and the 18-8 alloy vapor lines were low, a relatively thick scale of iron sulfide was formed on the carbon-steel wear plate.

In this particular case, it was postulated that, during the operation of the unit, beds of catalyst collected in the annular space between the wear plates and the 18-8 alloy vapor line. During the turnarounds, when the unit was steamed free of hydrocarbons, the beds of catalyst between the wear plates and 18-8 alloy vapor lines were wetted with water. After the unit had been opened, air was present for the oxidation of the iron sulfide to iron sulfite and sulfate. These salts hydrolyzed, forming sulfurous and sulfuric acids which attacked the 18-8 alloy—resulting in failure of the metal along the grain boundaries.

As a result of this work, the carbon-steel wear plates were removed from all 18-8 alloy tubes and vessels in the catalyst system, and wear plates of 18-8 alloy were installed. This eliminated the formation of iron sulfide between the wear plates and the 18-8 alloy lines and eliminated the intergranular corrosion of the 18-8 alloy line and vessels.

[The material presented in the concluding section of Mr. Camp's paper discusses the corrosion problems encountered in the vapor-phase reforming of a light naphtha. This data is omitted here since it has previously been published—"Stop Tube Failure in Superheater by Adding Corrosion Inhibitor," by E. Q. Camp, Cecil Phillips and Lewis Gross, *National Petroleum News Technical Section*, 38 (10) R-192 (1946).—The Editors.]

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PERSONALS

Petroleum Technologists in the Headlines

Dr. John R. Bates, formerly assistant to Sun Oil Co.'s vice president in charge of manufacturing, has been appointed Director of Research for the company and will head the newly-created Research Department. The creation of the independent Research Department is the first step in the eventual consolidation of most of the company's scientific activities into a proposed Science Center at Newtown Square, Pa., in a non-industrial area about 15 miles from the Marcus Hook refinery.

Dr. Bates

Mr. Hill

Mr. Kurtz

Dr. Bates will be assisted in his new work by Dr. J. Bennett Hill, now manager of the development division of the Manufacturing Department; **Dr. John M. Pearson,** now director of physical research and development of the Production Department, and **Stewart S. Kurtz, Jr.,** manager of the experimental division of the Manufacturing Department.

Dr. Bates received his A. B. degree from Amherst College in 1924 and was the recipient of three fellowships at Princeton, from which he received an M. A. in 1925 and Ph. D. in 1927. He was a national research fellow at Johns Hopkins, an international research fellow at the University of Berlin and a research associate at Princeton. He joined the staff of University of Michigan in 1932 as assistant and associate professor of general and physical chemistry.

In 1936 he joined the Catalytic Development Co. and became its director of research, resigning in 1942 to join the manufacturing department of Sun Oil Co.

Dr. J. Bennett Hill took both his undergraduate and graduate work at University of Pennsylvania and joined the Barrett Co. as research chemist, later becoming laboratory manager. In 1924 he became chief research chemist for Atlantic Refining Co., leaving in 1934 to join Sun Oil Co. as manager of its newly-formed Development Division at the Marcus Hook refinery.

Dr. John M. Pearson, a native of Oregon, attended Reed College in Portland and later the University of Chicago, where he received his B. S. degree in 1925. He served as an instructor at California Institute of Technology for three years, receiving a Ph. D. degree there in 1930, after he was already in the employ of Sun Oil Co. After serving with its pipe line affiliates, in 1945 he became director of Sun's physical laboratory near Chester, Pa.

Stewart S. Kurtz, Jr., received an A. B. from Harvard in 1921 and an M. A. degree in 1922 after military service in World War I. He served as a research chemist with Goodyear Rubber Co. and held a similar position with Atlantic Refining Co., resigning in 1935 to join Sun Oil Co. as manager of the Chemical Section of the Development Division at the Marcus Hook refinery.

* * *

Carl W. Georgi, Enterprise Oil Co., Buffalo, is stepping down as chairman of National Lubricating Grease Institute's technical committee after seven years as its head. Succeeding him will be another well-known technologist, **T. G. Roehner,** Socony-Vacuum Oil Co., Brooklyn, N. Y.

* * *

D. E. Carr, formerly Director of Research for Union Oil Co. of California, is now consultant on lubricants in the Research Department of Phillips Petroleum Co., Bartlesville, Okla.

* * *

Robert G. Atkinson, formerly with Hydrocarbon Research, Inc., is now a member of the chemical engineering staff of Phillips Petroleum Co.

* * *

William T. Guinee, of the electrical department at the Bayway refinery of Standard Oil Co. of New Jersey, has been made foreman of that department. **James McTernan** has been made head of the boilermaking department.

* * *

Dr. Donald G. Zink is superintendent of the chemical products department of Stanolind Oil & Gas Co., Tulsa. He was formerly director of technical development of United States Industrial Chemicals, Inc.

H. R. Lyles is senior process engineer on the staff of Cities Service Co. For the past 9 years he has been in the research and development laboratory of Lago Oil & Transport Co., at Aruba in the West Indies.

* * *

Dr. Dan Fore, Jr. is the newest addition to the Research Department staff at Phillips Petroleum Co., Bartlesville, Okla. He comes to Phillips from the Naval Research Laboratory at Washington, D. C., where he conducted and directed research on fuels for Navy use. Dr. Fore graduated from Mississippi College in Clinton, Miss., and obtained his Ph. D. in chemistry from the University of North Carolina.

Mr. Fore

* * *

Dr. Alex G. Oblad is Director of Chemical Research at the Houdry Process Corp. laboratories near Marcus Hook, Pa. He graduated and received his Ph. D, in physical chemistry from the University of Utah and was for a time in the research department of Standard Oil Co. (Indiana) and later with Magnolia Petroleum Co., where he became chief of chemical research. He was later head of industrial research for the Texas State Research Foundation at Dallas.

Dr. Oblad

Dr. Dart

Dr. Jack C. Dart is Director of Development at the Houdry laboratories. He received his A. B. degree from Albion College and his Master's degree in chemical engineering from University of Michigan. His professional career includes work with Pan-American Refining Corp., Magnolia Petroleum Corp. and Standard Oil Development Co.

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The author, Dr. Welcher is identified as associate professor of chemistry in the Extension Division, Indiana University. The last volume, No. IV, is expected to be published before the end of the year.

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The methods presented are arranged according to the type of reaction involved in the titration rather than by the field in which they may be applied. In this way the authors are able to emphasize the fundamental principles upon which each method is based, making the book an excellent text. On the other hand, those readers who wish to make use of it as a ready reference will find the extensive subject index a distinct help.

Senior author I. M. Kolthoff is Professor and Head of the Division of Analytical Chemistry at the University of Minnesota. V. A. Stenger is Analytical Research Chemist with Dow Chemical Co.

Copies of all books reviewed here may be ordered from the Reader's Service Department, Petroleum Processing, 1213 West Third St., Cleveland 13, for the prices listed, postpaid, unless indicated otherwise. Ohio purchasers please add 3% tax.

Distillation Literature Since 1920 Covered in German Bibliography

Bibliography of Distillation and Rectification, edited by H. Stage and G. R. Schultze, 5¼ x 8½ in., 186 pages, paper binding, author index, $6.75.

The *Bibliography of Distillation and Rectification* covers literature published between 1920 and 1944 on the theory of distillation and rectification, calculation and design of distillation columns, methods of operation, types of columns, and distillation problems. It is a reprint of the original German work, *Die Grundlegenden Arbeiten ueber Theorie, Apparate sowie Verfahren der Destillation und Rektifikation*, published in 1944 in Berlin.

The listing appears fairly complete and includes work from British, American, South American, Canadian, French, Dutch, Italian, Japanese, and Russian publications. Entries are made in the languages in which the original papers were published.

The introduction, orginally in German, has been translated into English, as has been the table of contents. However, the subheadings covering the various classifications are in German. Because of the languages involved, it would seem that the bibliography will have a limited use for American industry, especially those companies with limited facilities for ready translation.

The bibliography was listed in the Office of Technical Services' weekly report for June 27, 1947, as available in photostat and microfilm copies. Since that time, the copyright, vested in the U. S. Attorney General, has been licensed to a private concern, the Hobart Publishing Co., and the OTS is not selling copies.

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EDITORIAL

COMMENTS

WIDENING HORIZONS

It Rests with the Oil Companies To Develop Synthetic Fuels

THAT the oil companies will take the leadership in the development of methods for making fuels and lubricants from sources other than petroleum is indicated by the amounts they are spending for research as compared with other industries directly or indirectly interested in this subject. Data on research expenditures by these groups was gathered by the Federal Power Commission in its investigation of the natural gas industry. The FPC wanted to know what experimental work was being carried on to produce gas by synthesis to augment natural gas supplies when necessary.

The petroleum industry is expending at the rate of over $100,000,000 per year in research and development, exclusive of that for exploration for new crude supplies. This is about 3% of its total income. Aggregate expenditures for all industrial research in the U. S. are about $700,000,000 a year.

By comparison, the combined coal and coke industries spend barely $3,000,000 for research aside from that relating to improved materials handling equipment. The annual volume of anthracite and bituminous coal and coke products marketed is around 700,000,000 tons, nearly three times the volume of petroleum products. Research and development activities by the manufactured gas and natural gas utilities are even more limited.

Research work of the coal and gas groups in this field is limited to studies of synthetic gas production, leaving to the oil companies the further steps in the manufacture of liquid fuels and lubricants from either natural or synthesis gas.

Only a portion, of course, of the $100,000,000 a year the oil companies spend on research is for work on synthetic fuels. R. P. Russell, until recently president of Standard Oil Development Co., has stated that nearly one-third of the research and development effort of that company is directed to the conversion of natural gas and coal to liquid fuel. So it may well be that what the oil industry spends a year in this research is comparable with the $30,000,000 the Bureau of Mines is authorized to spend in five years on its so widely-heralded synthetic liquid fuels program.

The oil companies' research is continuous and of long standing and with important cumulative results, as was so dramatically demonstrated in the wartime aviation gasoline program. There is every reason to believe the achievement will be as great when their present work in synthetic fuels is translated into commercial output.

* * *

Along the same line of thought as the above, Marion E. Dice, Manager of the Economics Department, General Petroleum Corp. of California, told the annual meeting of the California Natural Gasoline Assn. there is no likelihood of a dying-off process where the petroleum industry will be replaced by some competing industry in supplying liquid fuels.

"The processes of the future are almost certain to remain in the hands of our own chemical engineers and their successors," he said. "Even the technical knowledge of today is sufficient, without any more developments, to provide our energy needs for many generations with materials already blocked out.

"But research has not stopped. In fact, the magnitude of the research investment is the most convincing proof that oil technology will not be superseded by coal. The petroleum industry spends 40 cents per ton of its product for research, excluding exploration research. The coal industry is reported to be spending 0.4 cents per ton. Which is likely to have the greatest reserve of technology 10 years hence?

The oil companies today, said Mr. Dice, are manufacturers of liquid fuels, not merely "refiners" of crude oil and natural gasoline. "We shall make the kind of fuels needed for the engines of the future, and in the quantity needed, without much regard to the source or nature of the raw material. Assuming free competition between materials, we shall use the cheapest material that will accomplish the job."

Why Professional Engineers Do Not Favor Labor Unions

AN enlightening comparison between the economic philosophies of the labor unions and of professional engineers in this country was presented by E. E. Rosaire, Houston, director of the National Society of Professional Engineers, speaking before a recent meeting of the American Society of Mechanical Engineers in that city.

The average union leader, said Dr. Rosaire, considers labor as a commodity like lands or goods, which he aims to sell in the highest price market while openly trying to rig the market by creating a monopoly—the closed shop. Next, the union boss aims to be a tight trader, giving as little as possible of his commodity for the agreed price. Finally, he sells his commodity on a lot basis, like a bushel of corn or a gallon of gasoline; or more like whiskey, Dr. Rosaire commented since the commodity, labor, is supposed to improve with age through establishing seniority.

However, the basic problems of this country have been those of an economy of abundance, the speaker brought out, and to that economy the professional engineer has contributed in a major way. If our national economy should become static, or worse, contract into one of scarcity, the engineers, chemists and other professional workers would be among the first to lose their jobs.

Thus, said Dr. Rosaire, the professional engineer realizes his economic welfare is highest in an expanding economy of abundance, which creates the greatest market for his services, and that anything which savors of an economy of scarcity, whether by action of capital, labor, management or government, is likely to adversely affect his personal interests. This fundamental difference in attitude towards society explains the great lack of success of the unions in bringing the professional workers into their fold.

"The professional engineers, quietly but openly and steadily", said the speaker, "have objected and protested against their being forced to join any organization with professed policies and preferred tactics which involve the basic idea of imposing an economy of scarcity, even though temporary, by stopping part of our national productive machinery".

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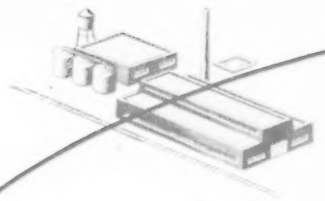

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EP — Equipment Patents
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Ed -- Editorial
EP -- Equipment Patents
Ht -- Helpful Trade Literature
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PT -- Patents Trends
T -- Tomorrow in Petroleum Technology
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